

# MISSOURI DEPARTMENT OF CONSERVATION

Spring River Sampling / Analysis

Fish and Wildlife Research Center • 1110 College Avenue

Columbia, Missouri 65201 • Phone 314/449-3761

LARRY R. GALE, Director

RECEIVED  
JAN 07 1986

Site: Syntex-Verona  
ID #: MO2007452154  
Break: 17.8  
Other: Spring River  
Sampling 9/19/85

SUPERFUND BRANCH

September 19, 1985

Mr. Scott Ritchey  
U.S. Environmental Protection Agency  
Region VII  
324 East Eleventh Street  
Kansas City, Missouri 64106



40033418  
SUPERFUND RECORDS

Dear Mr. Ritchey:

On August 5 and 6, 1985 white suckers (*Catostomus commersoni*) were collected from five locations on the upper Spring River. The fish were collected by electrofishing, wrapped in aluminum foil, frozen on dry ice and handled in a similar manner. Representatives from Syntex and the U.S. Environmental Protection Agency were present during sampling. The five sites corresponded to those identified in the "Verona Plant, Fish and Sediment Plan" with the exception of Site 4 which was moved .5 miles upstream. These sites were identical to those sampled in August of 1984. The sampling locations are identified in Attachment A. The size and weight of each fish and the identifying EPA code is listed in Attachment B. The recommended minimum numbers of fish were met at all locations.

The fish were taken to our facility at Columbia, Missouri, thawed and prepared accordingly. The fish at sites 2, 3, 4 and 5 were weighed and measured and sequentially placed into two equal size groups designated as Groups A and B. The right skinless fillets of the fish in Groups A and B were removed and placed in separate polyethylene bags. These two groups are to be analyzed separately. The remainder of Group B fish (the entire fish minus the right fillet) was placed in a third bag for analysis. A fourth whole body estimate will be calculated.\* The fish at Site 1 were prepared in a similar manner except they were sorted into three equal size groups. Groups A and B were prepared in a manner identical to sites 2-5 and the fish in Group C were

COMMISSION

JEFF CHURAN

JOHN POWELL

JOHN B. MAHAFFEY

RICHARD T. REED

Site: Syntex-Verona  
ID #: MD2007452154  
Break: 17.8  
Other: Spring River  
Sampling

Missouri Department Of Conservation

Mr. Scott Ritchey

-2-

September 19, 1985

simply left whole and refrozen for analysis. Thus a total of 16 composites were prepared for analysis which will generate 21 data points (five calculated).

The fish will be shipped to Dr. Mike Gross, University of Nebraska, by Federal-Express. A copy of this letter was forwarded to Dr. Gross.

Sincerely,

*Ron Crunkilton*

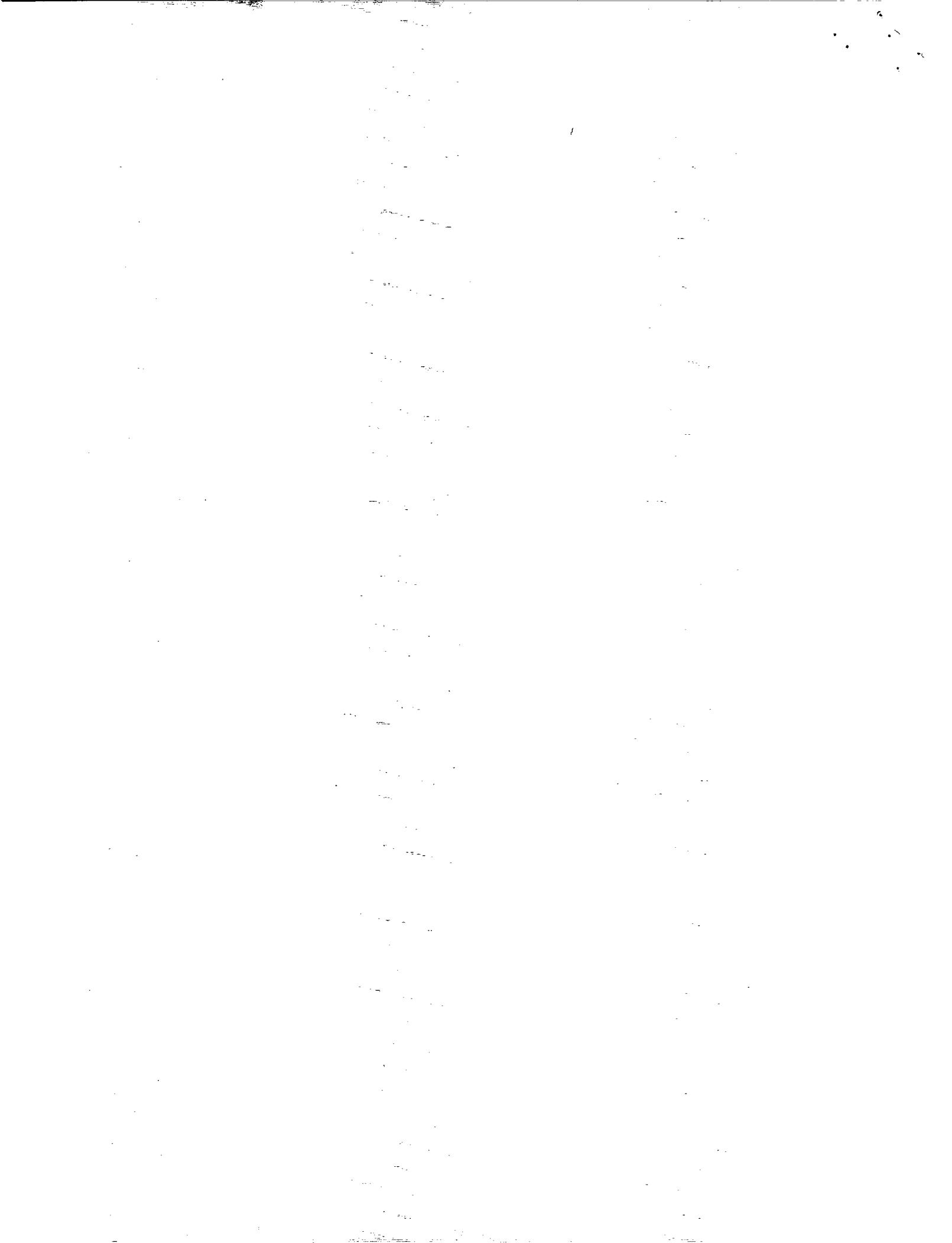
Ron Crunkilton, Biologist  
Water Quality Research

RC:aa

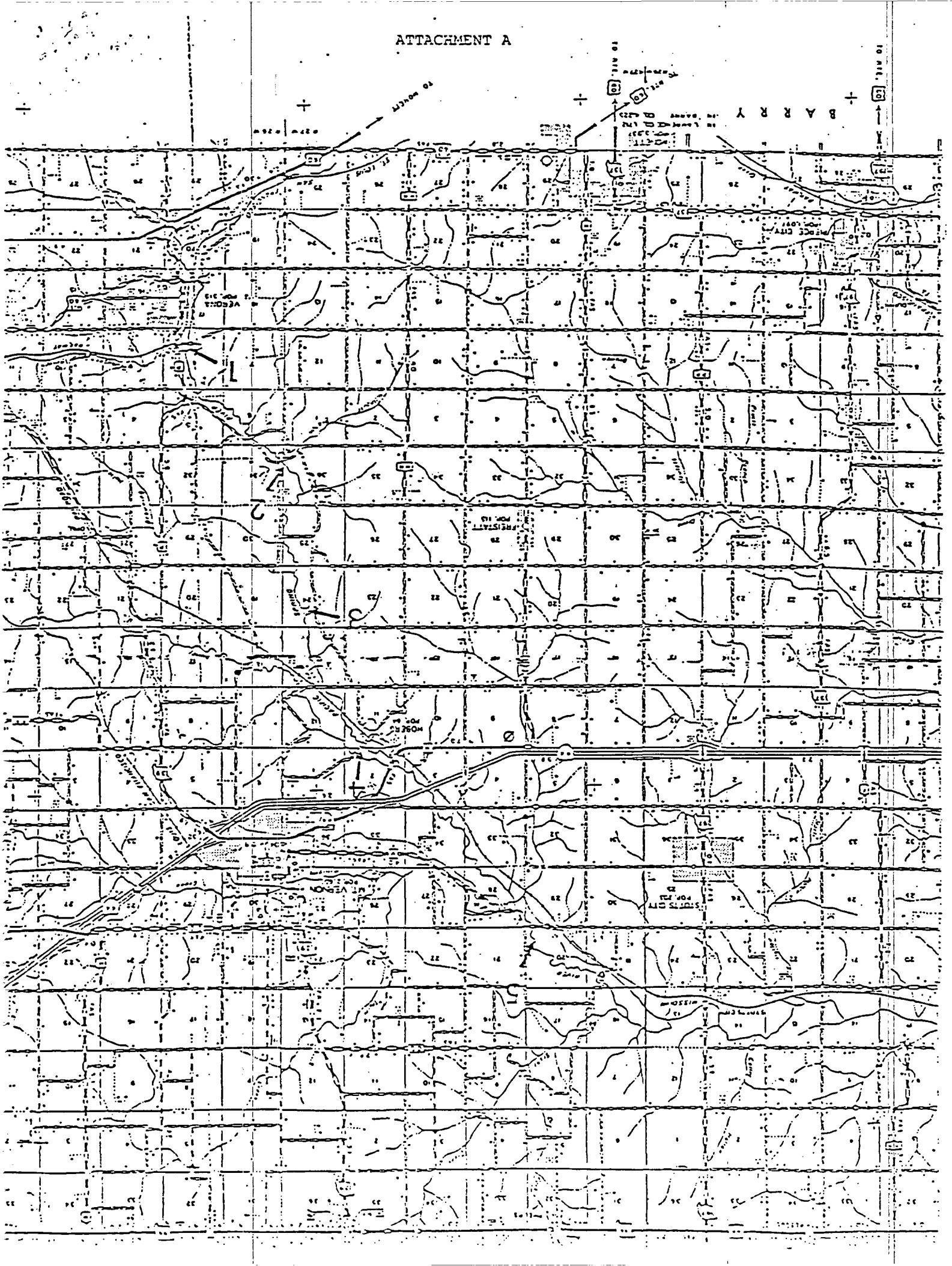
Enclosure

cc: Dr. David Robertson  
Dr. Gross

- \* These whole body values are to be determined from the weighted average of the Group B concentration (whole body minus right fillet) and Group B concentration (fillet only).



ATTACHMENT A



Attachment B

**Site 1 - Group A - Fillets Only**

| Total Length (mm) | Wt (kg) | EPA Number |
|-------------------|---------|------------|
| 331               | .350    | AKJC4-001  |
| 354               | .420    |            |
| 252               | .160    |            |
| 267               | .190    |            |
| 272               | .200    |            |
| 288               | .236    |            |
| 317               | .359    |            |
| 412               | .845    |            |

**Site 1 - Group B - Fillets and Remainder**

| Total Length (mm) | Wt (kg) | EPA Number                    |
|-------------------|---------|-------------------------------|
| 320               | .330    | AKJC4-002 (fillets)           |
| 258               | .169    | and                           |
| 366               | .569    | AKJC4-003 (remainder)         |
| 276               | .230    | and                           |
| 271               | .200    | AKJC4-004* (to be calculated) |
| 342               | .387    |                               |
| 313               | .335    |                               |
| 240               | .185    |                               |

**Site 1 - Group C - Whole Fish**

| Total Length (mm) | Wt (kg) | EPA Number |
|-------------------|---------|------------|
| 253               | .154    | AKJC4-005  |
| 260               | .183    |            |
| 299               | .268    |            |
| 335               | .364    |            |
| 275               | .193    |            |
| 264               | .201    |            |
| 309               | .297    |            |
| 360               | .459    |            |

**Site 2 - Group A - Fillets Only**

| Total Length (mm) | Wt (kg) | EPA Number |
|-------------------|---------|------------|
| 318               | .305    | AKJC4-006  |

\* Total Wt of fillets for Group 1B fish is .350 kg for calculation purposes.

Site 2 - Group A - Fillets Only (continued)

| Total Length (mm) | Wt (kg) | EPA Number |
|-------------------|---------|------------|
| 366               | .551    |            |
| 423               | .903    |            |
| 320               | .324    |            |
| 375               | .544    |            |

Site 2 - Group B - Fillets and Remainder

| Total Length (mm) | Wt (kg) | EPA Number                    |
|-------------------|---------|-------------------------------|
| 378               | .501    | AKJC4-007 (fillets)           |
| 386               | .446    | and                           |
| 344               | .387    | AKJC4-008 (remainder)         |
| 323               | .351    | and                           |
| 297               | .286    | AKJC4-009* (to be calculated) |
| 307               | .314    |                               |

Site 3 - Group A - Fillets Only

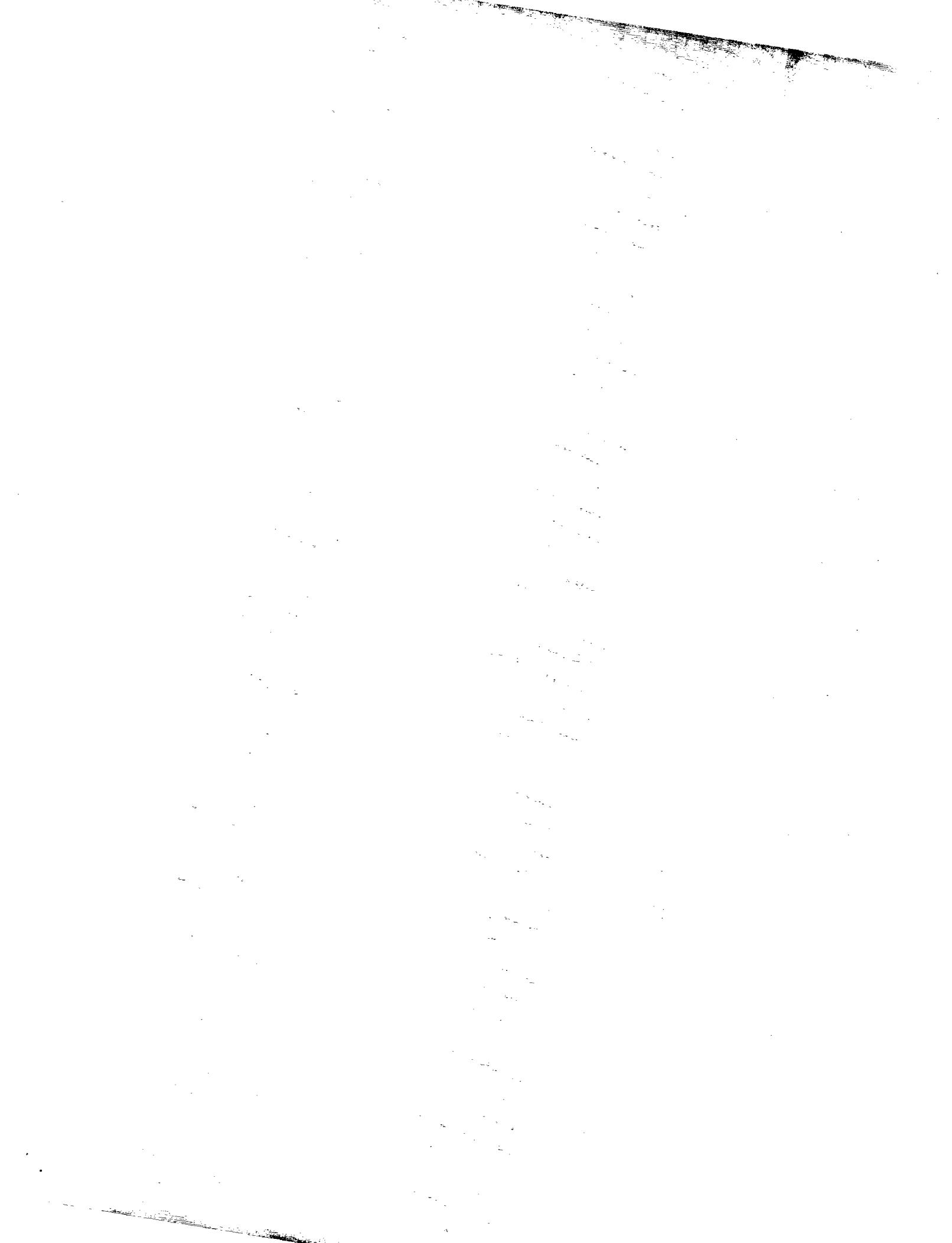
| Total Length (mm) | Wt (kg) | EPA Number |
|-------------------|---------|------------|
| 344               | .411    | AKJC4-010  |
| 306               | .284    |            |
| 332               | .374    |            |
| 326               | .339    |            |
| 357               | .460    |            |
| 287               | .236    |            |
| 287               | .263    |            |
| 394               | .645    |            |

Site 3 - Group B - Fillets and Remainder

| Total Length (mm) | Wt (kg) | EPA Number                    |
|-------------------|---------|-------------------------------|
| 318               | .344    | AKJC4-011 (fillets)           |
| 294               | .250    | and                           |
| 349               | .432    | AKJC4-012 (remainder)         |
| 302               | .287    | and                           |
| 300               | .381    | AKJC4-013* (to be calculated) |
| 392               | .797    |                               |
| 340               | .393    |                               |
| 373               | .599    |                               |

\* Total weight of fillets for Group 2B fish is .412 kg for calculation purposes.

\* Total weight of fillets for Group 3B fish is .510 kg for calculation purposes.



Site 4 - Group A - Fillets Only

| Total Length (mm) | Wt (kg) | EPA Number |
|-------------------|---------|------------|
| 255               | .180    | AKJC4-014  |
| 315               | .298    |            |
| 341               | .442    |            |
| 358               | .533    |            |
| 446               | .977    |            |

Site 4 - Group B - Fillets and Remainder

| Total Length (mm) | Wt (kg) | EPA Number                    |
|-------------------|---------|-------------------------------|
| 254               | .149    | AKJC4-015 (fillets)           |
| 278               | .229    | and                           |
| 349               | .420    | AKJC4-016 (remainder)         |
| 346               | .470    | and                           |
| 398               | .589    | AKJC4-017* (to be calculated) |

Site 5 - Fillets Only

| Total Length (mm) | Wt (kg) | EPA Number |
|-------------------|---------|------------|
| 360               | .535    | AKJC4-018  |
| 298               | .270    |            |
| 379               | .578    |            |
| 321               | .366    |            |
| 457               | 1.006   |            |
| 311               | .332    |            |

Site 5 - Fillets and Remainder

| Total Length (mm) | Wt (kg) | EPA Number                    |
|-------------------|---------|-------------------------------|
| 335               | .378    | AKJC4-019 (fillets)           |
| 297               | .279    | and                           |
| 291               | .257    | AKJC4-020 (remainder)         |
| 367               | .476    |                               |
| 371               | .662    |                               |
| 456               | .901    | AKJC4-021* (to be calculated) |

\* Total weight of fillets for Group 4B fish is .210 kg for calculation purposes.  
\* Total weight of fillets for Group 5B fish is .448 kg for calculation purposes.

RECEIVED

OCT 01 1985

September 19, 1985

CMPL SECTION

MEMORANDUM

SUBJECT: Trip Report - Spring River Sediment Sampling, Verona, Missouri

FROM: ✓ Paul E. Doherty  
Chief, SINV/EP&R/ENSV

TO: ✓ Robert L. Morby  
Chief, SPFD

THRU: William J. Keffer  
Chief, EP&R/ENSV

John C. Wicklund  
Director, ENSV

David A. Wagoner  
Director, HSTM

Attached for your information is a trip report on the sediment sampling conducted at the above referenced site.

If you have any questions or comments, please call me at 236-3888.

Attachments

✓cc: Charles Hensley, LABO

sent Q-34-RS

CONCURRENCES

|         |   |         |              |  |  |  |  |  |  |  |  |
|---------|---|---------|--------------|--|--|--|--|--|--|--|--|
| SYMBOL  | H | PLD     | CONCURRENCES |  |  |  |  |  |  |  |  |
| SURNAME | H | 9/19    |              |  |  |  |  |  |  |  |  |
| DATE    | H | 1/19/85 |              |  |  |  |  |  |  |  |  |



# ecology and environment, inc.

FAIRWAY WEST OFFICE BLDG., 4250 SHAWNEE MISSION PARKWAY, SHAWNEE MISSION, KS 66205, TEL. 913-432-9961

International Specialists in the Environment

TO: Paul Doherty, ARPO  
FROM: Karen Koth, FIT  
DATE: September 10, 1985  
SUBJECT: Trip Report of Sampling Activities at Spring  
River Basin (Syntex)  
TDD # R-07-8508-04A  
Sample Series: AKJC4

On August 27, 1985, sediment sampling was completed at 3 stations (Station 1, Station 3, and Station 5) downstream from the Syntex Agribusiness Plant in Verona, Missouri (See Attachment 1). This is the second year of sampling, of a five-year plan, completed under the Verona Plant Fish and Sediment Plan (Syntex Plan). The Syntex plan consists of a Consent Agreement and Order between Syntex Agribusiness, Inc. and the U.S. Environmental Protection Agency (EPA). Contained in this report is information concerning the sediment sampling portion of the work plan. Fish sampling by the Missouri Department of Conservation (MDC) was completed August 5-6, 1985, at all five stations along the Spring River. The sample series, AKJC4, was also used for these samples. Further information about the fish sampling can be obtained from Ron Crunkleton of MDC (314-449-3781).

The following persons participated in the sediment sampling effort:

Karen Koth, FIT  
Bob Wiggins, FIT  
Bill Durbin, Syntex Representative

Sediment sampling at the 3 stations was completed as follows:

### Station 1

Location: 0.3 miles downstream from Syntex (junction of Spring River and "Farm Road.")

Spring River Basin  
Page 2

Sample: Sediment - 10 aliquots across the stream bed

Time of Collection: 1145 hrs.

Collected by: Wiggans, Koth

Depth of Collection: 0-6"

Method: Using a deconned shovel, sediment was transferred to a 2000 micron sized opening, sieve. The sieved sediment was then collected in a stainless-steel pan where it was blended prior to transfer to separate laboratory containers.

Assigned Sample Number: AKJC4023

Size of Sample Container: 1 - 16 oz. glass jar with teflon-lined lid (2 - 16 oz. jars were provided for Syntex split)

Station 3

Location: 6 miles downstream from Syntex - access gained via Mr. Buehler's property (RFD 2, Verona (417) 678-2284)

Sample: Sediment - 10 aliquots across the stream bed

Time of Collection: 1030 hrs.

Collected by: Wiggans, Koth

Depth of Collection: 0-6"

Method: Using a deconned shovel, sediment was transferred to a 2000 micron sized opening, sieve. The sieved sediment was then collected in a stainless-steel pan where it was blended prior to transfer to separate laboratory containers.

Assigned Sample Number: AKJC4022

Size of Sample Container: 1 - 16 oz. glass jar with teflon-lined lid (2 - 16 oz. jars were provided for Syntex split).

Station 5

Location: 12 miles downstream from Syntex (junction of Spring River and State Highway Y).

Sample: Sediment - 10 aliquots across the stream bed

Time of Collection: 0900 hrs.

Collected by: Wiggans, Koth

Depth of Collection: 0-6 inches

Method: Using a deconned shovel, sediment was transferred to a 2000 micron sized opening, sieve. The sieved sediment was then collected in a stainless-steel pan where it was blended prior to transfer to seprate laboratory containers.

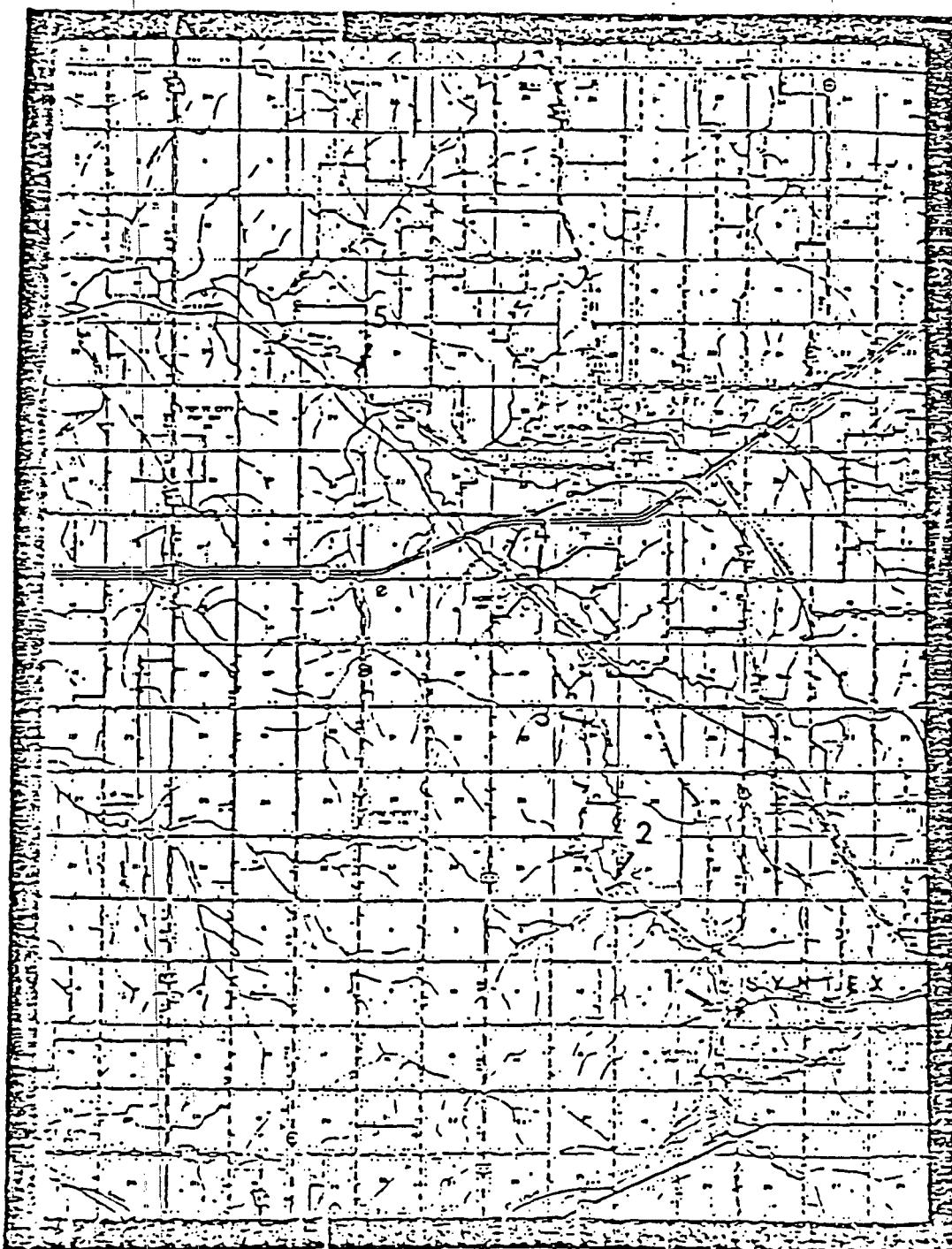
Assigned Sample Number: AKJC4021

Size of Sample Container: 1 - 16 oz. glass jar with teflon lined lid (2 - 16 oz. jars were provided for Syntex split).

Sediment samples were transported to the Region VII Laboratory on August 28, 1985, and chain of custody records were maintained with EPA personnel. Splits of the 3 sediment samples were turned over to Syntex representative Bill Durbin in the field at about 1300 hrs., August 27, 1985.

KK:nw

CENTRAL HIGHWAY MAP  
LAWRENCE COUNTY  
MISSOURI



MISSOURI STATE HIGHWAY DEPARTMENT  
DIVISION OF PLANNING

IN COOPERATION WITH THE  
US DEPARTMENT OF TRANSPORTATION  
FEDERAL HIGHWAY ADMINISTRATION

Sediment samples taken at

locations 1, 3 and 5

Client: U.S. EPA

Ref No.: R-07-8508-04A

Camera: Pole Nikon 35mm

Date:

Photographer: Koth

Date/Time: 8/27/85 1200hrs

Lens: Type:

SN:

Frame No.: 5

Comments: Location 1



Photographer: Koth

Date/Time: 8/27/85 1100hrs

Lens: Type:

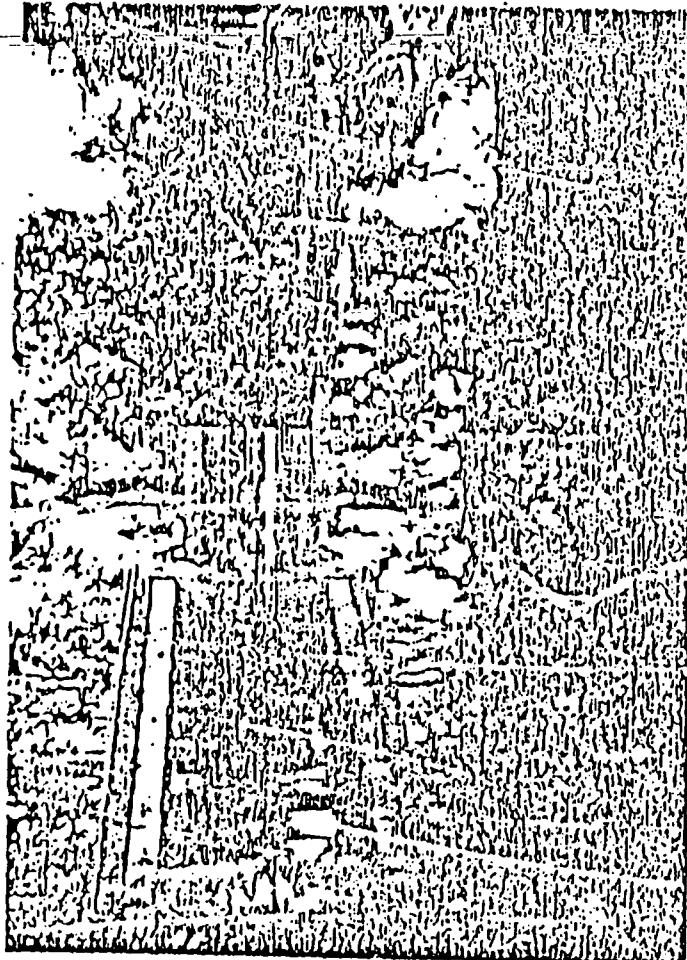
SN:

Frame No.: 4

Comments: Location 3

PHOTOGRAPHIC RECORD

Client: U.S. FPA      Lab Job No.: B-07-8508-000  
Camera: Polaroid      Date: 8/27/85



Photographer: Koth  
Date/Time: 8/27/85 0930hrs  
Lens: Type: \_\_\_\_\_  
SN: \_\_\_\_\_  
Filter No.: 2  
Comments: Location 5

Photographer: \_\_\_\_\_  
Date/Time: \_\_\_\_\_  
Lens: Type: \_\_\_\_\_  
SN: \_\_\_\_\_  
Filter No.: \_\_\_\_\_  
Comments: \_\_\_\_\_



University of  
Nebraska  
Lincoln

Department of Chemistry  
Hamilton Hall  
Lincoln, NE 68588-0304  
402-472-3501

RECEIVED

December 4, 1985

JAN 07 1986

Dr. Lewis Throop  
SYNTEX RESEARCH  
3401 Hillview Avenue  
Palo Alto, CA 94304

SUPERFUND BRANCH

Dear Lew:

Enclosed is a report "Analysis of 2,3,7,8-Tetrachlorodibenzo-p-dioxin in Fish Samples" for our 1985 analytical activities for you. Also enclosed are copies of the mass profiles obtained during the elution time of 2,3,7,8-TCDD. Please note that one sample gave us a problem with recovery. We are repeating the extraction and analysis and will report it to you as part of an addendum.

We will submit a bill to you when the repeat analysis is complete and the results accepted by you.

Please let me know if there are any problems and what we are to do with the left-over samples.

Best wishes,

  
Michael L. Gross  
Professor of Chemistry

MLG:lm  
c.c: Dr. N.C.A. Weerasinghe  
Enclosure

RECEIVED

DEC 10 1985

DR. L. THROOP

JAN 07 1986

Report

SUPERFUND BRANCH

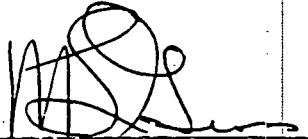
Analysis of 2,3,7,8-Tetrachlorodibenzo-p-dioxin in Fish Samples

for

Syntex Agribusiness, Inc.

by

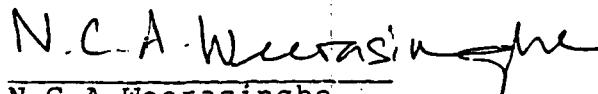
Michael L. Gross  
Midwest Center for Mass Spectrometry  
Department of Chemistry  
University of Nebraska-Lincoln  
Lincoln, NE 68588



Michael L. Gross  
Professor & Director

12-02-85

Date



N.C.A. Weerasinghe  
Senior Research Associate

TCDDREP52

Sample Extraction Procedure

The fish fillets, filleted fish and the whole fish samples were ground by using a fish grinder and mixed thoroughly in order to make homogenous samples. Approximately 20g aliquots of ground fish from each sample was taken and fortified with the internal standard,  $^{13}\text{C}_{12}$ 2,3,7,8-TCDD (2.5ng). A 40% solution of KOH and alcohol (15ml) was added, and the mixture was refluxed for about 1 hr. to saponify the tissue. The resulting solution was extracted with hexane (3 x 30ml) and the hexane extracts were combined. The combined hexane extract was washed with conc. sulfuric acid followed by water and concentrated under a stream of nitrogen. The concentrated extracted (ca 1ml) was subsequently submitted to liquid chromatography cleanup.

Liquid Chromatography Clean-Up.Silica Chromatography

Silica for chromatography was used without any activation. A 5 cm column was prepared in a disposable pipette and the silica was capped with a 1/4 cm layer of anhydrous sodium sulfate. The column was washed with hexane (ca. 5 ml) prior to transferring the sample extract. The extract was quantitatively transferred, and eluted with hexane (4 ml) followed by 20% benzene in hexane (5 ml). The eluates were combined and concentrated while benzene was being replaced with

hexane for alumina chromatography.

### Alumina Chromatography

Alumina was washed by saturating with methylene chloride, removing excess solvent, then activating at 165°C for 24 hours. A 5 cm column was prepared in a disposable pipette and capped with a 1/4 cm layer of anhydrous sodium sulfate. The column was cooled to room temperature in a dessicator before use.

Hexane was used to wet the column before transferring the sample. The jar was rinsed with one ml of hexane which was transferred to the column. The alumina was eluted with 6 ml of pesticide grade  $\text{CCl}_4$ , then with 4 ml of 10%  $\text{CH}_2\text{Cl}_2$  and finally with 5 ml of 25%  $\text{CH}_2\text{Cl}_2$  in hexane. The methylene chloride/hexane fraction was collected and concentrated under nitrogen while replacing the volatile  $\text{CH}_2\text{Cl}_2$  with hexane. All other fractions were discarded.

### Carbon Chromatography

Carbon (Amoco PX-21) for chromatography was obtained from Amoco Corporation. Carbon was mixed with previously washed (in a Soxhlet with methanol) silica gel (2g of silica and 50mg of carbon) and packed in disposable pipets. A disposable pipet was cut at the narrow end (end A), and a disc cut out of a glass fiber filter paper was used as a plug at the other end (end B). The pipet was then filled with a 1/2 cm layer of silica and 2 cm layer a carbon/silica mixture. The column was then capped with a 1/4 cm layer of anhydrous sodium sulfate. The column was then washed in the direction B to A with methylene chloride (5ml), 1:1 methylene chloride/benzene (5ml) and toluene (5ml). Subsequently, it was washed in the A to B direction with hexane (5ml), and the eluant from the alumina chromatography

(evaporated and redissolved in methylene chloride) was transferred onto the column. It was then eluted in succession with methylene chloride (5ml), 1:1 methylene chloride/benzene (5ml) and finally, 2,3,7,8-TCDD was eluted in the reverse flow (B to A) with toluene. The toluene eluant was evaporated under a stream of dry nitrogen to a final volume of ca. 0.05ml and use for analysis.

List of Materials Used in Sample Extraction

Acetone, OmniSolv, MCB

Carbon tetrachloride, OmniSolv, MCB

Ethyl alcohol, OmniSolv, MCB

Hexane, OmniSolv, MCB, non UV

Methylene chloride, OmniSolv, MCB

Sulfuric acid, concentrated, analytical reagent, Mallinckrodt

Water, distilled in glass

Potassium hydroxide, analytical grade, Mallinckrodt

Sodium sulfate (anhydrous), analytical grade, Fisher

Sodium carbonate (anhydrous), analytical grade, Fisher

Aluminum oxide, neutral, activity grade I, Woelm Pharma

Dry nitrogen (boil-off from liquid N<sub>2</sub>)

All OmniSolv line solvents are distilled in glass, suitable for chromatography and residue analysis.

### Isomer Specific TCDD Analysis by Capillary Column GC/HRMS

Appropriate dilutions of the samples were made with hexane at the time of analysis and the aliquots from the resulting solutions were used for capillary column GC/HRMS.

#### Gas Chromatography/Mass Spectrometer

A Kratos MS-50 high resolution mass spectrometer (ultimate resolution 150,000), equipped with a custom-built multiple peak monitoring (MPM) device was used. The mass spectrometer was coupled to a Carlo-Erba Gas chromatograph. Two capillary columns were used during the analysis. An SP 2340 (0.32mmx30m) fused silica capillary column was used in the initial screening of the samples. Subsequently, the sample extracts were chromatographed on SP 2330 (0.32mmx60m) fused silica capillary column.

#### Gas Chromatographic Conditions

Typical operating conditions were: Helium with a linear velocity of  $\sqrt{45}$  cm/sec, injector 250°C, detector 260°C. The GC parameters were: column temperature 70°C, isothermal for 3 minutes, and then programmed at 30°/min to 250°C. An on column injection technique was utilized. The sample was injected in hexane at a temperature of 70°C and programmed to 250°C.

#### Mass Spectrometric Conditions and Multiple Ion Selection

The mass spectrometer was operated in the EI mode (70eV, 250°C) at 10,000 resolving power. The ions m/z 319.8965, m/z 321.8936 and

m/z 333.9339 ( $^{13}\text{C}_{12}$ -2,3,7,8-TCDD) were monitored on three channels using the MPM. The instrument was tuned using m/z 330.9792 of PFK, and this ion was used as a check mass on channel 4. Mass profiles were acquired by using computer controlled custom-built MPM at an amplifier bandwidth of 1000Hz and a sweep width and time of 300ppm and 5 Hz respectively. A real time chromatogram was stored during the GC/MS run which was later recalled to extract necessary mass profiles for the peak that corresponds to 2,3,7,8-TCDD.

#### Calculation of Results

The concentration of 2,3,7,8-TCDD was calculated using the 'internal standard ratio method'. An average ratio for normalized signal responses for masses 334 and 322 for  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD and native 2,3,7,8-TCDD, respectively, were obtained by injecting standards throughout the analysis. The ratio of signal responses for masses 322 and 334 in samples was compared with the average ratio for standards in order to determine the concentration. The detection limit was considered to be 2.5 X the noise amplitude for each sample.

The internal standard,  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD was utilized in the calculation of percent recoveries, and in doing so, the absolute intensity of m/z 334 in samples was measured and compared with an average value of intensities for m/z 334, obtained by injecting the internal standard.

#### Validation

The theoretical isotope abundance ratio for m/z 320/322 is 0.77. Positive signals for m/z 320 and 322 are considered to be valid

if the response ratio falls within  $0.77 \pm 0.1$ .

Comments

The samples, Site-2B (fillet and remainder) and Site 4A (fillet), had positive signals for both ions m/z 320 and 322. However, the isotope abundance ratio, m/z 320/322 that is used to confirm any positive signals for TCDD was higher than the theoretical ( $0.77 \pm 0.1$ ) ratio. The levels of 2,3,7,8-TCDD are low in these two samples. On the basis of the mass profiles, an interference giving an ion of mass near to m/z 319.8965, causes the intensity at 319.8965 to be elevated. Nevertheless, we cannot confirm the positive detection of 2,3,7,8-TCDD in these two samples.

The sample, Site-5B (whole fish), had poor recovery (< 5%) of the internal standard. Another aliquot of this sample will be reextracted, reanalyzed, and reported as an addendum to this report.

Analysis of Fish Fillet, Filleted Fish and Whole Fish  
for 2,3,7,8-TCDD

| Sample ID                         | Concentration<br>ppt | Detection<br>Limit<br>ppt | 320/322<br>Isotope<br>Ratio | Percent<br>Recovery |
|-----------------------------------|----------------------|---------------------------|-----------------------------|---------------------|
| Site 1A (Fillet)                  | 4.5                  | 1.0                       | .75                         | 85                  |
| Site-1B (Fillet<br>and Remainder) | 3                    | 1.0                       | .76                         | 60                  |
| Site-1C (Whole Fish)              | 16                   | 2.5                       | .85                         | 65                  |
| Site-2A (Fillet)                  | 3                    | 1.5                       | .86                         | 50                  |
| Site-2B (Fillet<br>and Remainder) | 3                    | 1.0                       | .95                         | 80                  |
| Site-2B (Whole Fish)              | 13<br>14+            | 7<br>5                    | .85<br>.87                  | 50                  |
| Site-3A (Fillet)                  | 1.5                  | 1.0                       | .62                         | 30                  |
| Site-3B (Fillet<br>and Remainder) | ND                   | 1.5                       |                             | 65                  |
| Site-3B (Whole Fish)              | 7                    | 1.5                       | .81                         | 45                  |
| Site-4A (Fillet)                  | 1.3                  | 0.9                       | .94                         | 50                  |
| Site-4B (Fillet<br>and Remainder) | 1<br>1+              | 0.7<br>0.9                | .87<br>.75                  | 75                  |
| Site-4B (Whole Fish)              | 6                    | 2.0                       | .81                         | 50                  |
| Site-5 (Fillet)                   | ND                   | 0.6                       |                             | 65                  |
| Site 5 (Fillet<br>and Remainder)  | ND                   | 0.5                       |                             | 80                  |
| Site-5 (Whole Fish)               |                      | To be reextracted.        |                             |                     |

Notes:

Approximately 20g of sample was fortified with 2.5ng of  $^{13}\text{C}_{12}$ -2,5,7,8-TCDD as the internal standard for recovery measurement and quantification.

The recovery of the internal standard for Sample Site 5 (whole fish) was less than 5%. This sample will be reextracted and reanalyzed.

\* Repeat analysis

**Appendix to Report**

**Analysis of 2,3,7,8-Tetrachlorodibenzo-p-dioxin  
in Fish Samples**

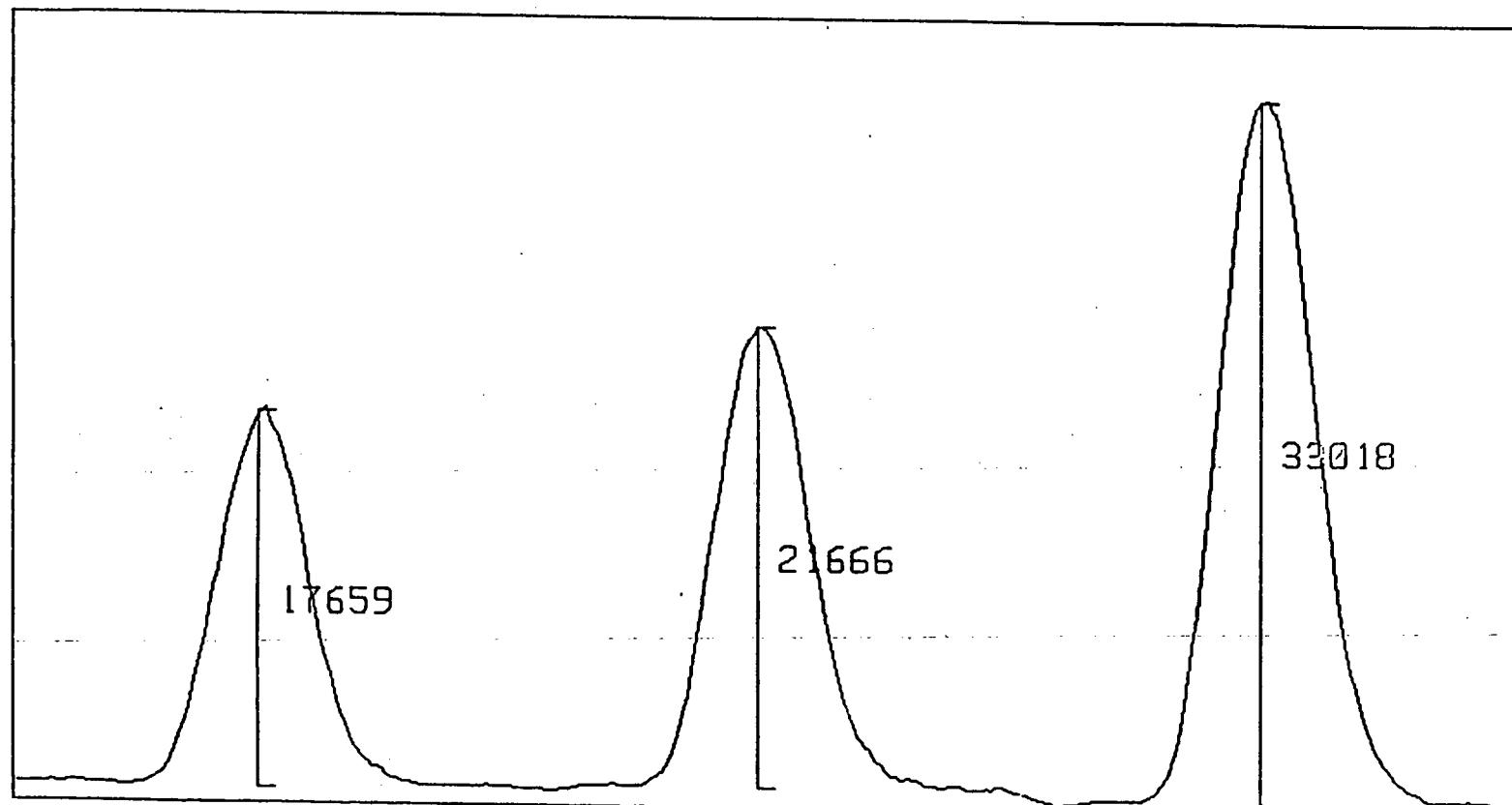
**December 2, 1985**

Standards

MCMS DIOX IN ANALYSIS 17: 7:13 11/19/85 T10252.DX  
RETN TIME: 8.47

WEIGHT RATIO: 14.00/ 100.00 # SCANS: 25  
TCDD/C-13 TCDD/TCDF/C-13 TCDF, 14/100/20/100 PG

SENSITIVITY = 82545. CNTS/NG  
SLOPE = 0.53  
RATIO = 0.815



| MASS   | 319.897 | 321.894 | 333.934 |
|--------|---------|---------|---------|
| GA IN  | 10.00   | 10.00   | 4.00    |
| 100%   | 37.099  | 37.099  | 37.099  |
| NO ISE | 2968.   | 2820.   | 3005.   |

Standards

MCMS DIOXIN ANALYSIS 12:49:53 11/21/85 T10287.DX

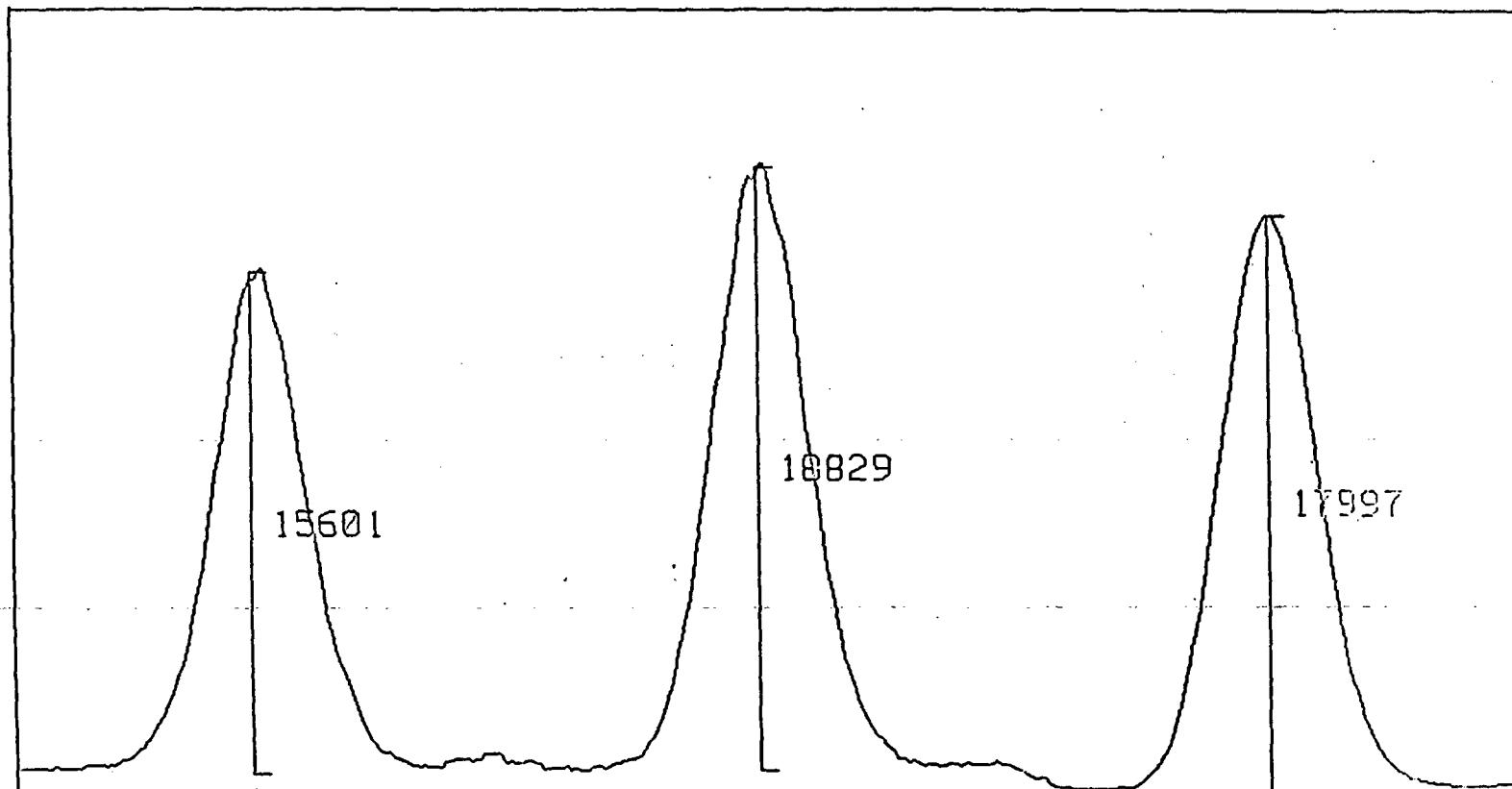
RETN TIME: 8.66

WEIGHT RATIO: 14.00/ 100.00 # SCANS: 18  
TCDD/C-13 TCDD/TCDF/C-13 TCDF, 14/100/20/100 PG

SENSITIVITY = 71990. CNTS/NG

SLOPE = 0.54

RATIO = 0.829

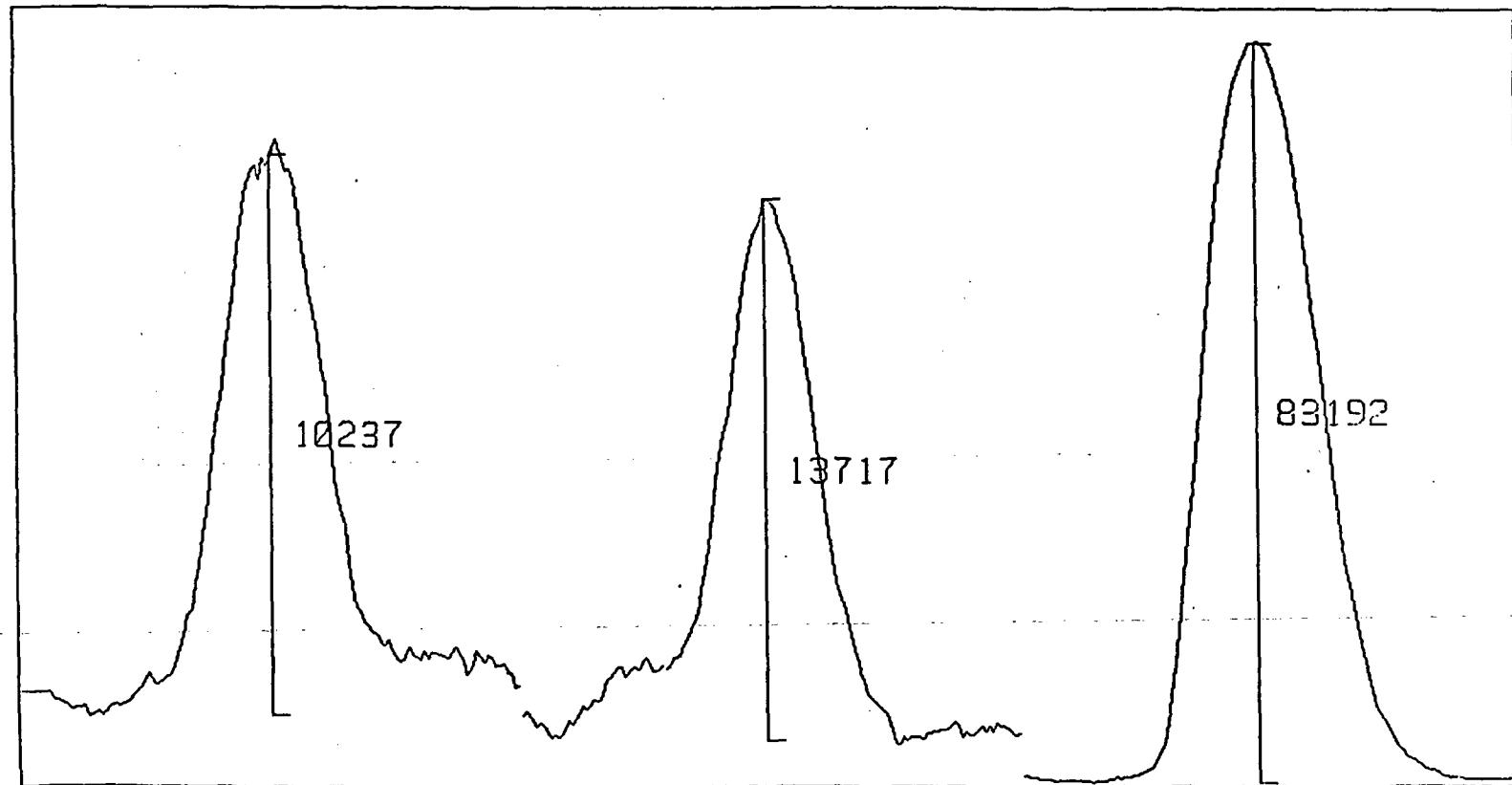


|       |         |         |         |
|-------|---------|---------|---------|
| MASS  | 319.897 | 321.894 | 333.934 |
| GAIN  | 10.00   | 10.00   | 2.50    |
| 100%  | 24453.  | 24453.  | 24453.  |
| NOISE | 2560.   | 2858.   | 1517.   |

Site 1A (Fillet)

MCM'S DIOXIN ANALYSIS    23: 8:26    11/19/85    T10259.DX  
RETN TIME: 8:18  
VOLUME RATIO: 5.00/ 41.00    # SCANS: 19  
SYNTEX FILLET, AKJC4-001 SITE 1A

DET. LIMIT = 1.2 PPT  
% RECOVERY = 83.34%  
CONCENTRATION = 4.6 PPT  
RATIO = 0.746

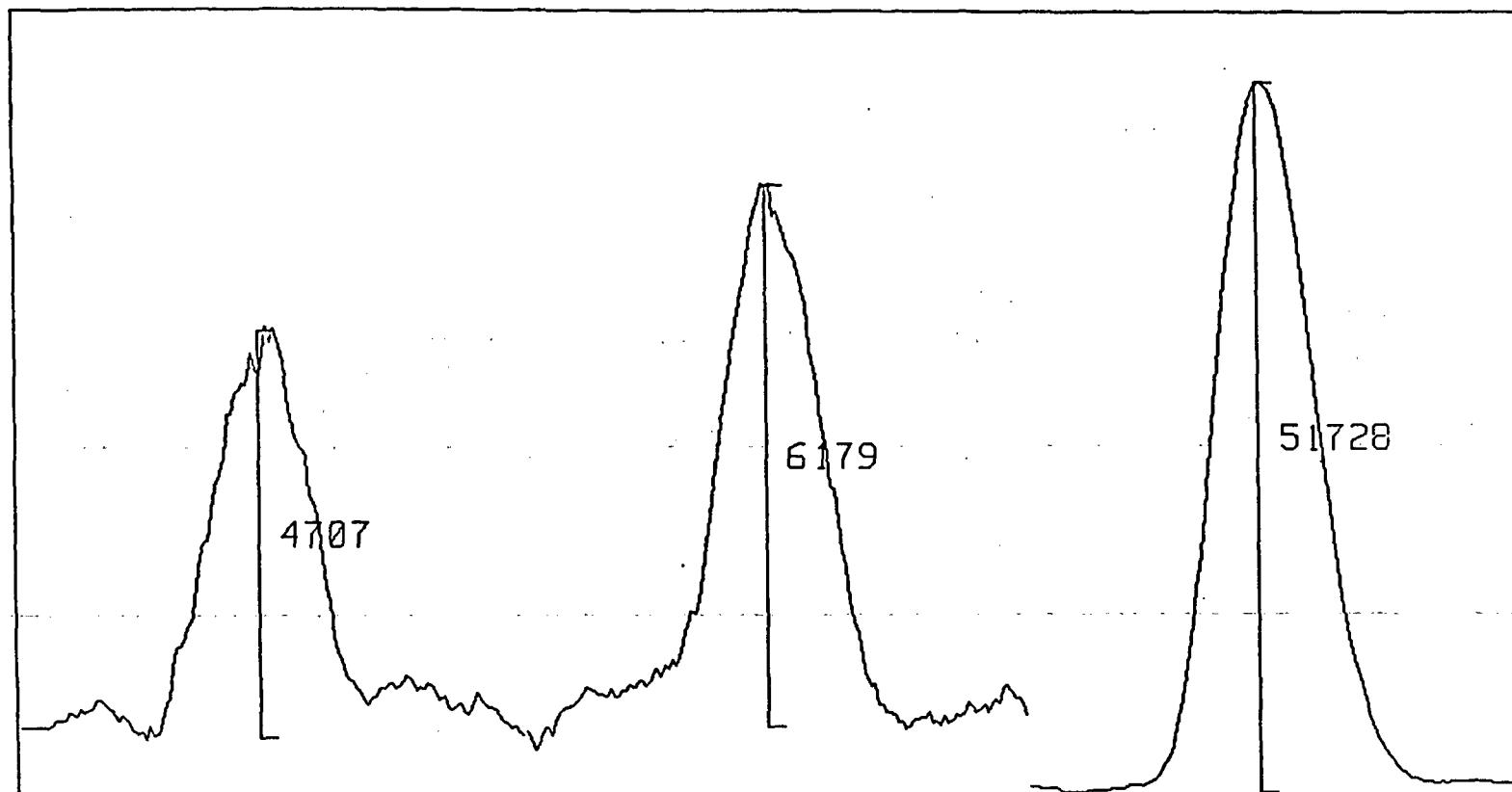


|       |         |         |         |
|-------|---------|---------|---------|
| MASS  | 319.897 | 321.894 | 333.934 |
| GAIN  | 10.00   | 10.00   | 4.00    |
| 100%  | 14257   | 19765   | 87570   |
| NOISE | 2841.   | 3602.   | 7333    |

Site 1B (Fillet +  
Remainder)

MCMS DIOXIN ANALYSIS    18:33:20    11/19/85    T10250.DX  
RETN TIME: 8.19  
VOLUME RATIO: 5.00 / 44.00    # SCANS: 21  
SYNTEX FILLET, AKJC4-002 SITE 1B

DET. LIMIT = 1.0 PPT  
% RECOVERY = 59.68%  
CONCENTRATION = 3.1 PPT  
RATIO = 0.762

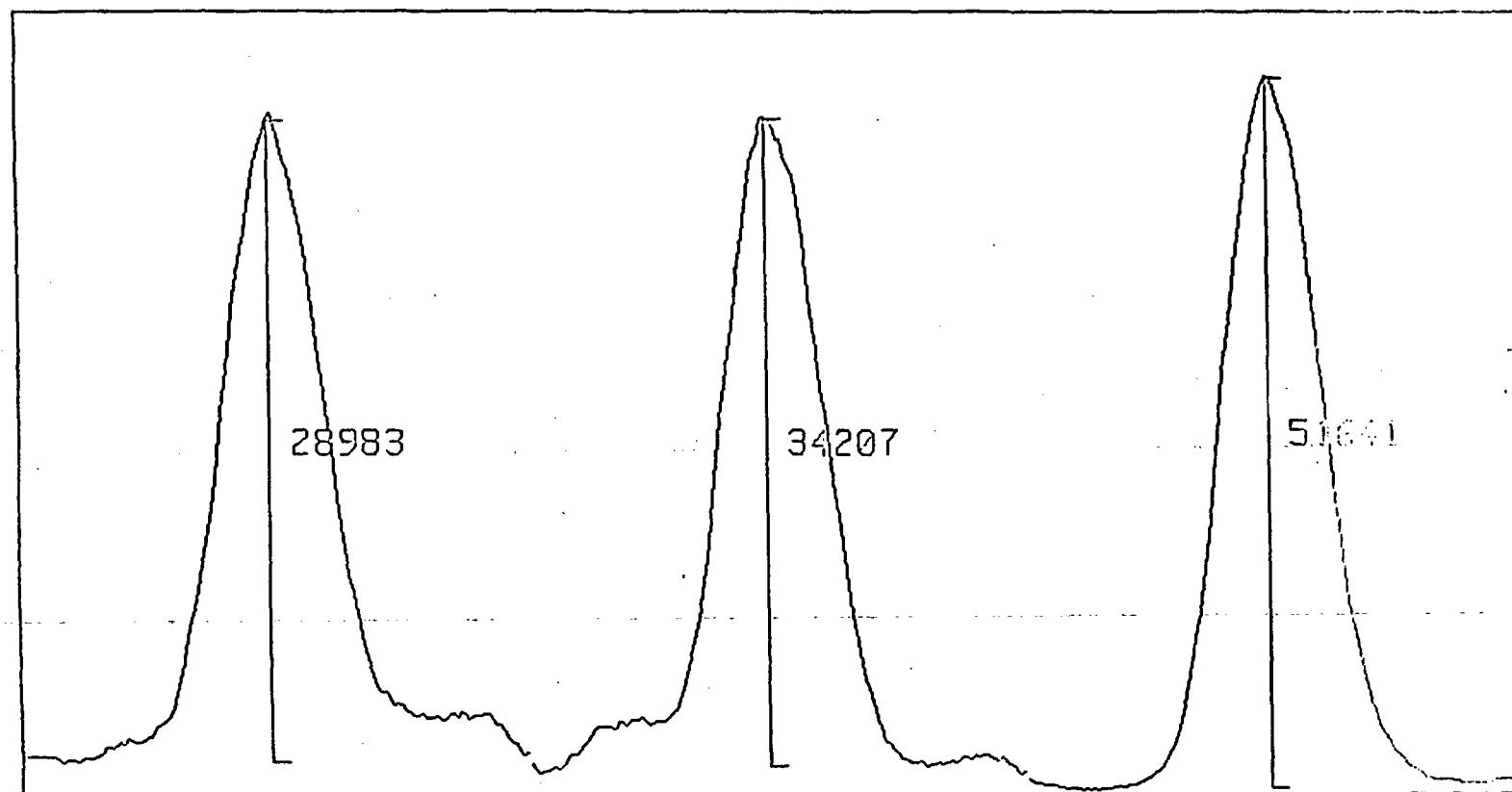


|        |         |         |         |
|--------|---------|---------|---------|
| MASS   | 319.897 | 321.894 | 333.934 |
| GAIN   | 10.00   | 10.00   | 4.00    |
| 100%   | 9086.   | 8981.   | 57348.  |
| NO ISE | 2245.   | 1873.   | 4233.   |

Site 1c (whole fish)

MCMS DIOXIN ANALYSIS 15:54:4 11/21/85 T10284.DX  
RETN TIME: 8.69  
VOLUME RATIO: 5.00/ 41.00 # SCANS: 17  
SYNTEX WHOLE FISH, AKJC4-003 SITE 1B

DET. LIMIT = 2.4 PPT  
% RECOVERY = 62.86%  
CONCENTRATION = 16.3 PPT  
RATIO = 0.847

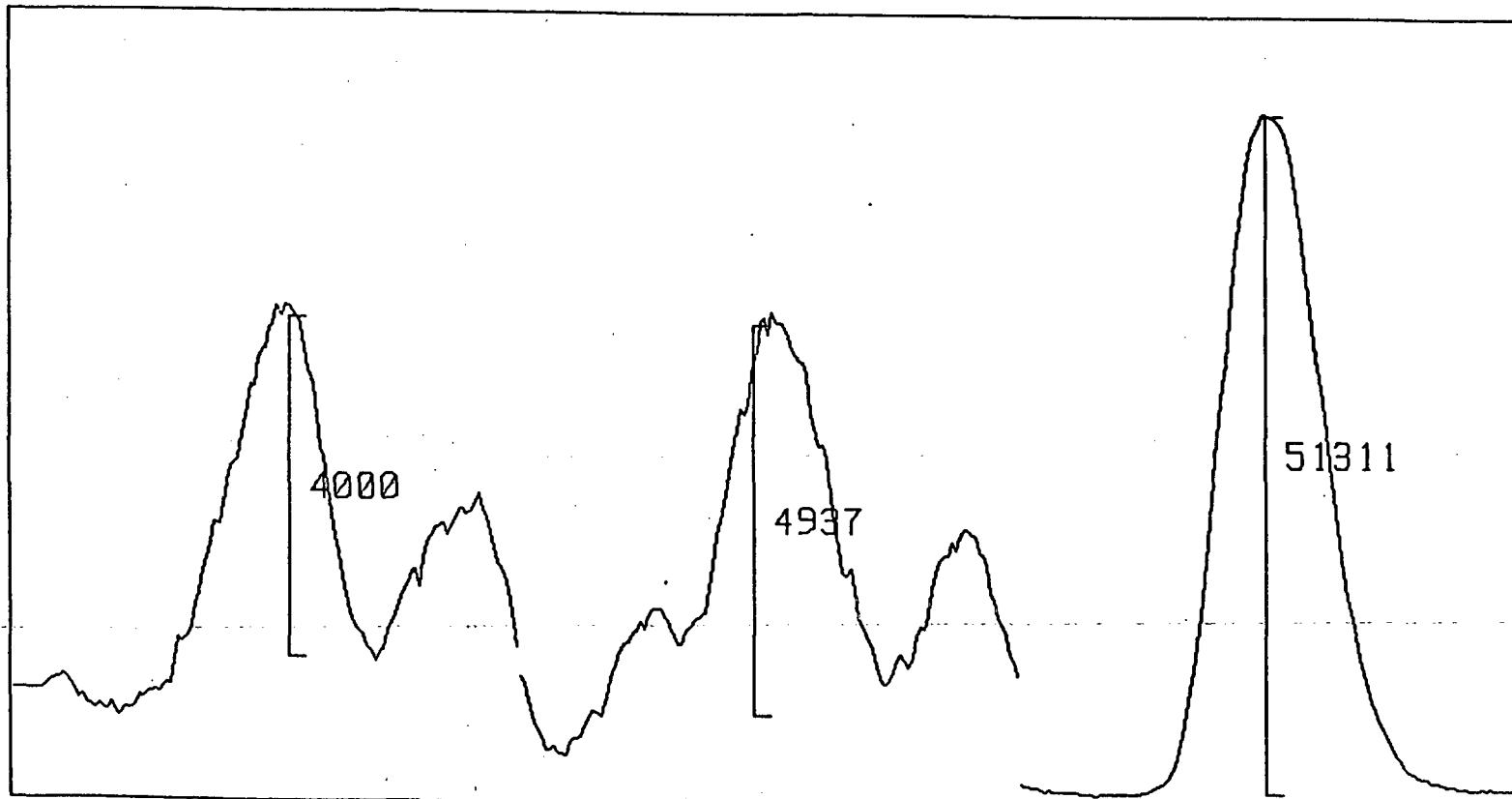


|       |         |         |         |
|-------|---------|---------|---------|
| MASS  | 319.897 | 321.894 | 333.934 |
| GAIN  | 10.00   | 10.00   | 4.60    |
| 100%  | 35518   | 41413   | 57125   |
| NOISE | 4537    | 5014    | 4319    |

Site 2A (Final)

MCMS DIOXIN ANALYSIS    22:42:48    11/19/85    T10258.DX  
RETN TIME: 8.32  
VOLUME RATIO: 5.00/ 39.00    # SCANS: 16  
SYNTEX FILLET, AKJC4-006 SITE 2A

DET. LIMIT = 1.4 PPT  
% RECOVERY = 50.27%  
CONCENTRATION = 2.9 PPT  
RATIO = 0.810

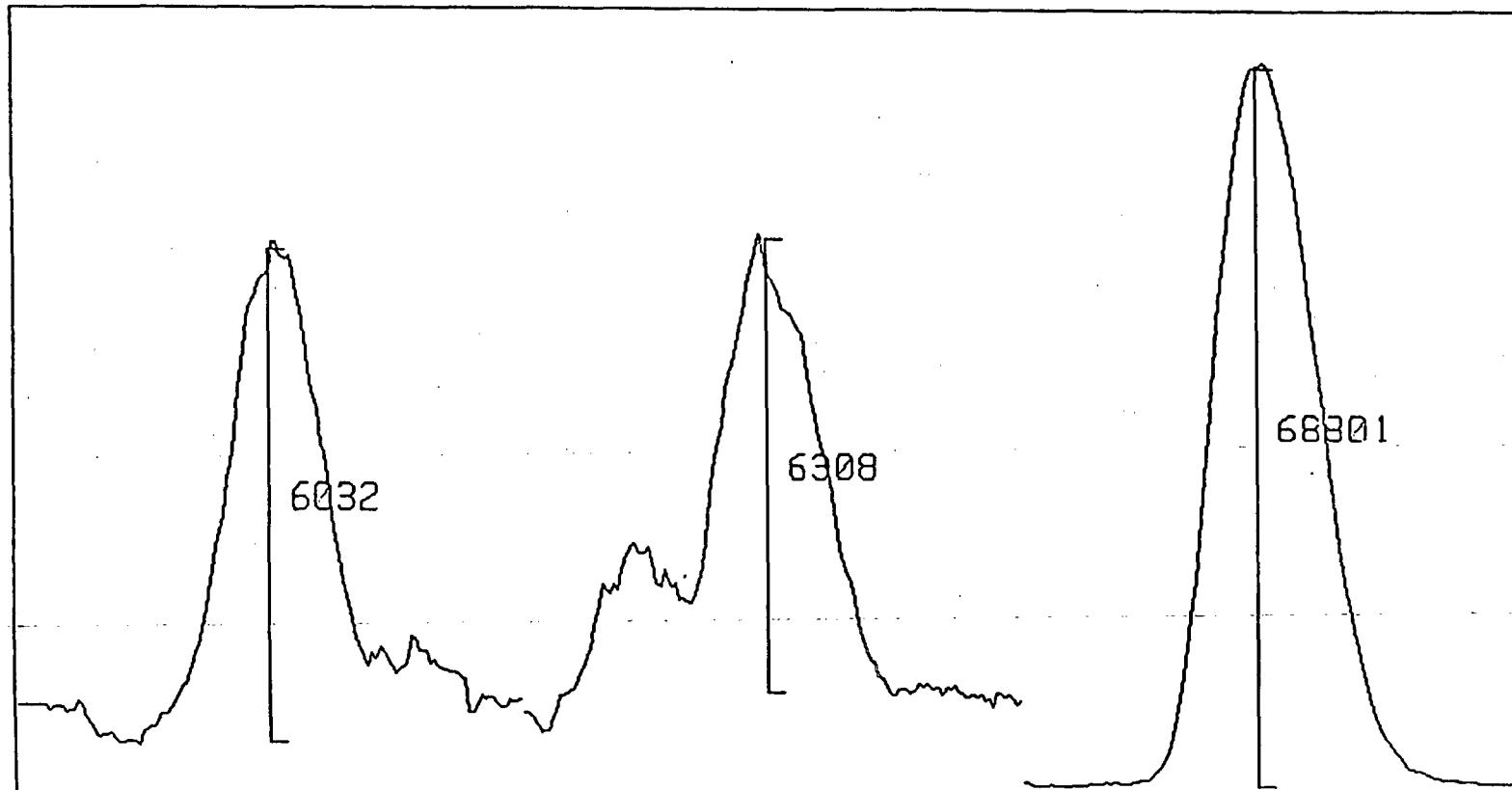


|        |         |         |         |
|--------|---------|---------|---------|
| MASS   | 319.897 | 321.894 | 333.934 |
| GA IN  | 10.00   | 10.00   | 4.00    |
| 100%   | 9345.   | 9993.   | 59803.  |
| NO ISE | 2026.   | 2361.   | 4824.   |

Site 2B (Fillet +  
Remainder)

MCMS DIOXIN ANALYSIS    19:21:23    11/19/85    T10251.DX  
RETN TIME: 8.22  
VOLUME RATIO: 5.00 / 45.00    # SCANS: 21  
SYNTEX FILLET, AKJC4-007 SITE 2B

DET. LIMIT = 1.1 PPT  
% RECOVERY = 81.18%  
CONCENTRATION = 2.7 PPT  
RATIO = 0.956

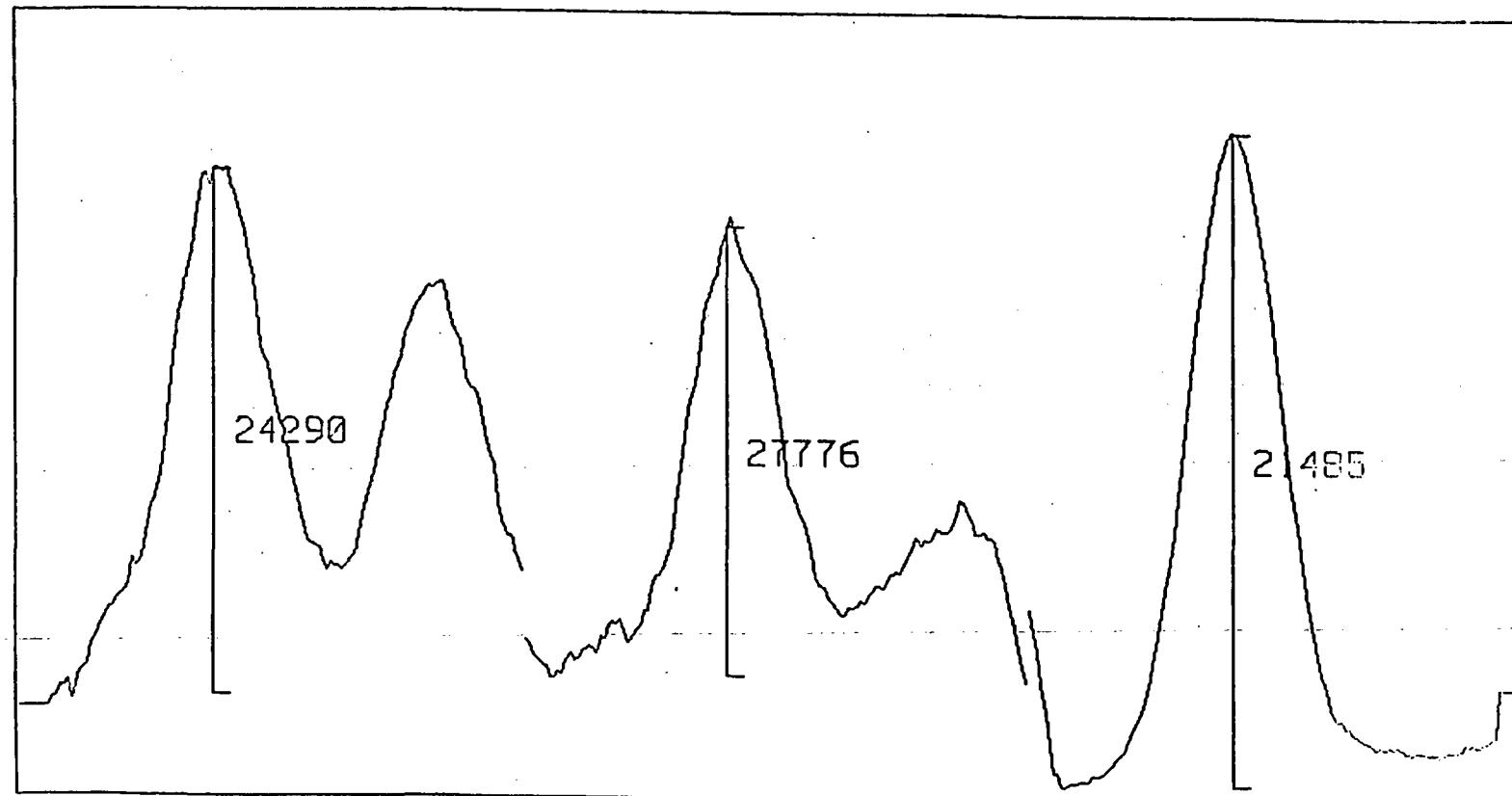


|        |         |         |         |
|--------|---------|---------|---------|
| MASS   | 319.897 | 321.894 | 333.934 |
| GA IN  | 10.00   | 10.00   | 4.00    |
| 100%   | 9636.   | 10952.  | 75275.  |
| NO ISE | 2161.   | 2499.   | 5529.   |

Site 2B (whole Fish)

MCMS DIOXIN ANALYSIS    11:50:24    11/22/85    T10293.DX  
RETN TIME: 8.71  
VOLUME RATIO: 7.00 / 50.00    # SCANS: 35  
VOLUME ADJUST.: 0.882  
SYNTEX FISH AKJC4-008 SITE 2B, RERUN

DET. LIMIT = 5.0 PPT  
% RECOVERY = 50.93%  
CONCENTRATION = 14.4 PPT  
RATIO = 0.875

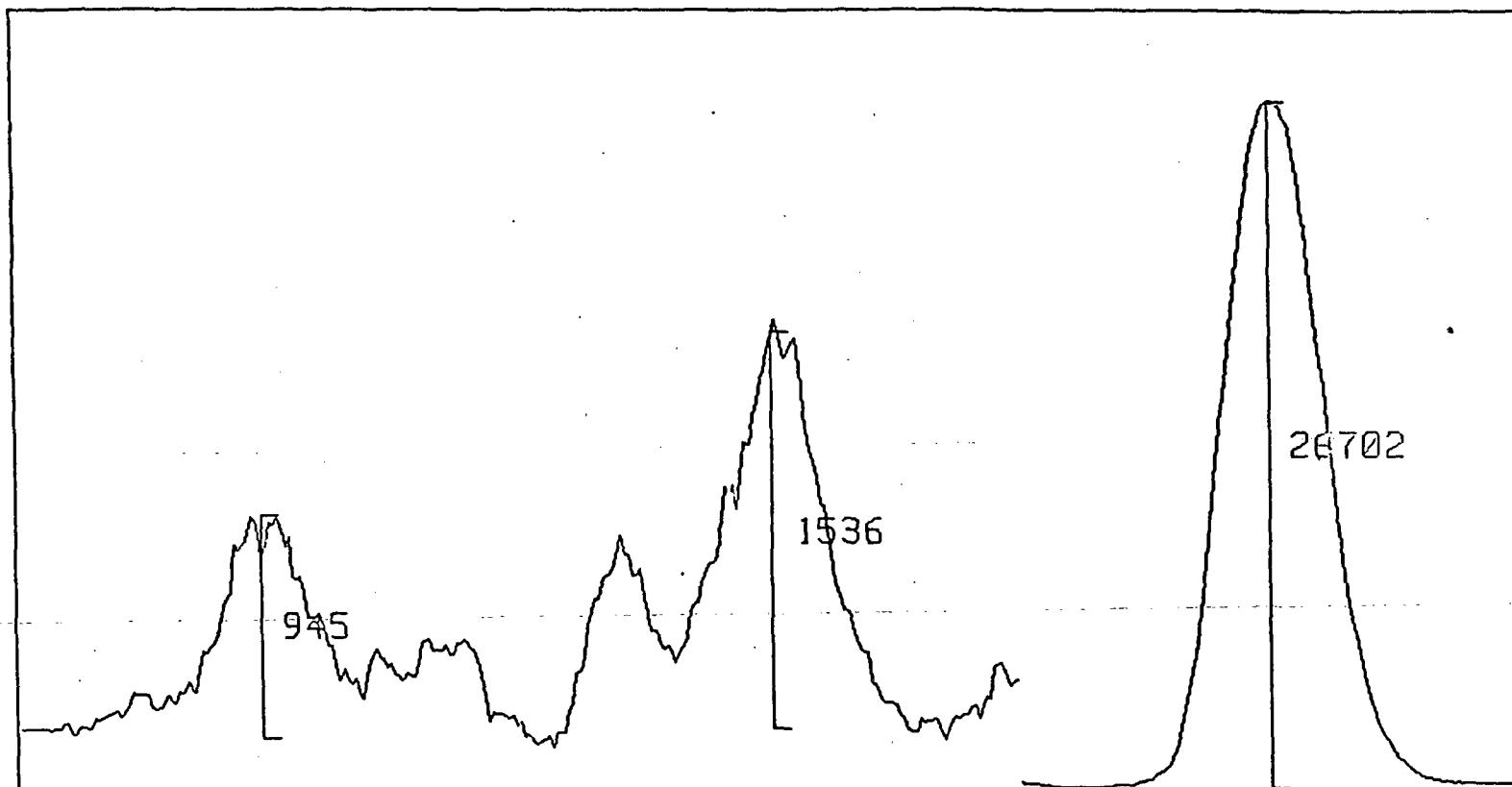


|       |         |         |         |
|-------|---------|---------|---------|
| MASS  | 319.897 | 321.894 | 333.934 |
| GA IN | 10.00   | 10.00   | 2.00    |
| 100%  | 36472   | 48559.  | 25885.  |
| NOISE | 8044.   | 9720.   | 2244.   |

Site 3A (Fillet)

MCMS DIOXIN ANALYSIS    20:36:53    11/19/85    T10255.OX  
RETN TIME: 8.29  
VOLUME RATIO: 5.00 / 39.00    # SCANS: 16  
SYNTEX FILLET, AKJC4-010 SITE 3A

DET. LIMIT = 1.0 PPT  
% RECOVERY = 27.30%  
CONCENTRATION = 1.6 PPT  
RATIO = 0.615

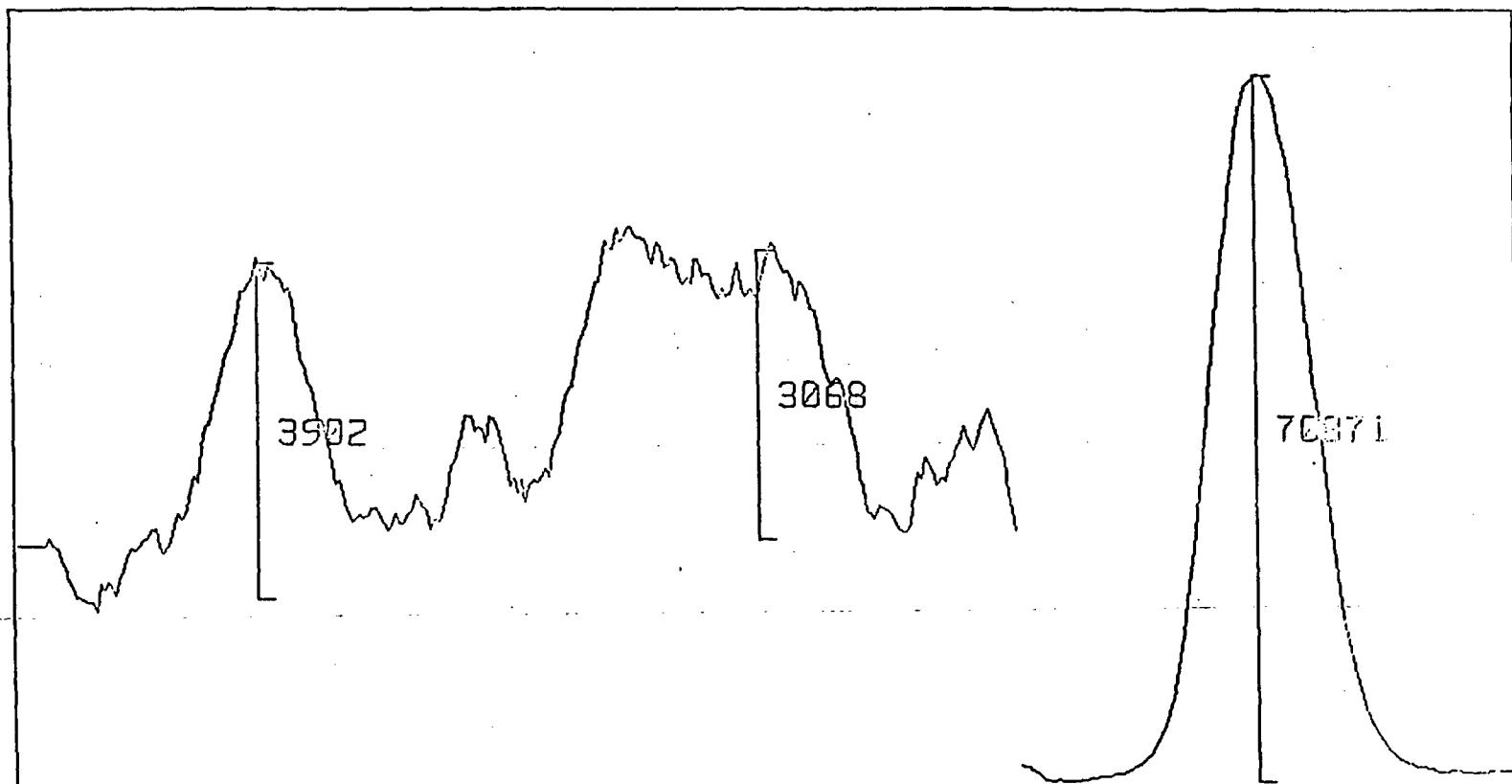


|        |         |         |         |
|--------|---------|---------|---------|
| MASS   | 319.897 | 321.894 | 333.934 |
| GAIN   | 10.00   | 10.00   | 4.00    |
| 100%   | 3327    | 3048    | 30482   |
| NO ISE | 990.    | 974.    | 2442.   |

Site 3B (Fillet +  
Remainder)

MCMS DIOXIN ANALYSIS    19:19:31    11/21/85    T10288.DX  
RETN TIME: 8.55  
VOLUME RATIO: 5.00/ 37.00    # SCANS: 18  
VOLUME ADJUST.: 0.914  
SYNTEX FILLET AKJC4-011 SITE 3B, RERUN

DET. LIMIT = 1.2 PPT  
% RECOVERY = 67.23%  
CONCENTRATION = 1.4 PPT  
RATIO = 1.272

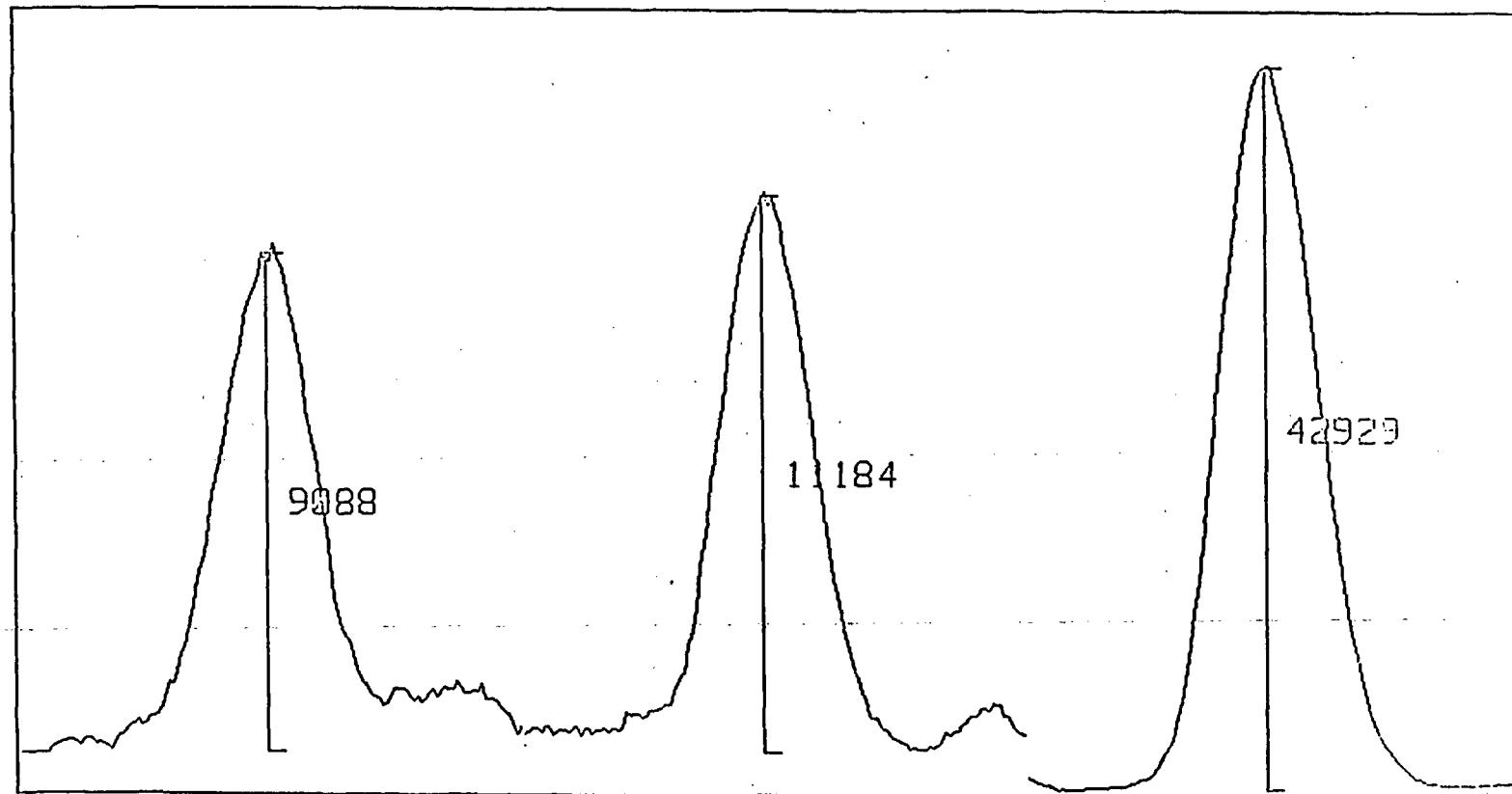


|       |         |         |         |
|-------|---------|---------|---------|
| MASS  | 319.897 | 321.894 | 333.934 |
| GAIN  | 10.00   | 10.00   | 5.00    |
| 100%  | 9075    | 8291    | 78052   |
| NOISE | 2456    | 2695    | 5506    |

Site 3B (whole Fish)

MCMS DIOXIN ANALYSIS    14:42:50    11/21/85    T10282.DX  
RETN TIME: 8.79  
VOLUME RATIO: 5.00/ 38.00    # SCANS: 12  
SYNTEX WHOLE FISH AKJC4-012 SITE 3B

DET. LIMIT = 1.6 PPT  
% RECOVERY = 46.57%  
CONCENTRATION = 7.0 PPT  
RATIO = 0.813



|       |         |         |         |
|-------|---------|---------|---------|
| MASS  | 319.897 | 321.894 | 333.934 |
| GAIN  | 10.00   | 10.00   | 4.00    |
| 100%  | 14379   | 15796   | 46662   |
| NOISE | 2366    | 2557    | 3638    |

Site 4A (Fillet)

MCMS DIOX IN ANALYSIS

11: 6:49

11/21/85 T10277.DX

RETN TIME: 6.83

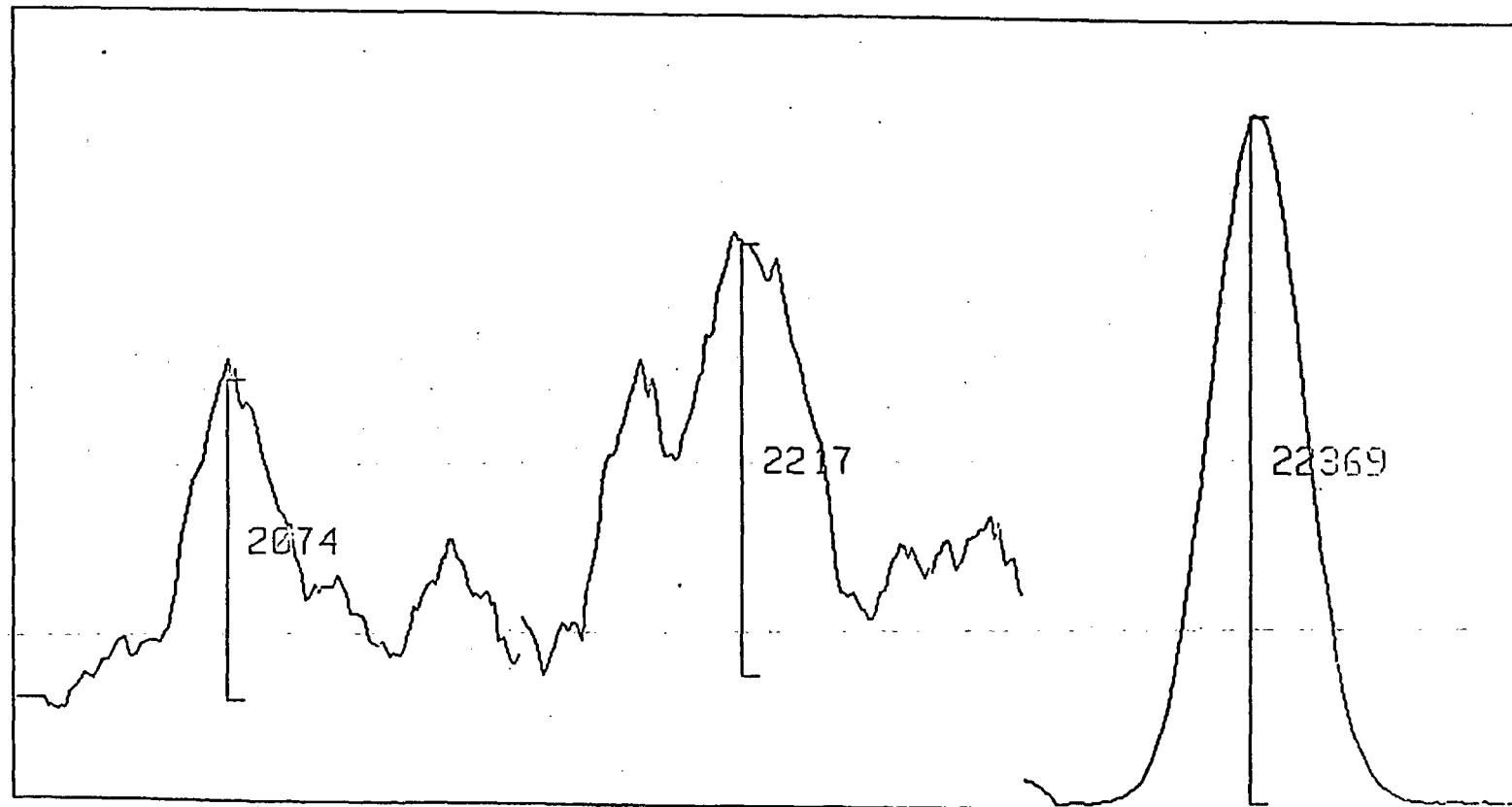
VOLUME RATIO: 4.80% 45.00

# SCANS: 22

VOLUME ADJUST: 0.875

AKC-014 SITE 4A (FILLET)

DET. LIMIT = 0.9 PPT  
% RECOVERY = 51.92%  
CONCENTRATION = 1.3 PPT  
RATIO = 0.935



MASS

319.897

GA IN

10.00

100%

51.33

NO ISE

1464

321.894

10.00

4061

1458

333.934

2.00

25712

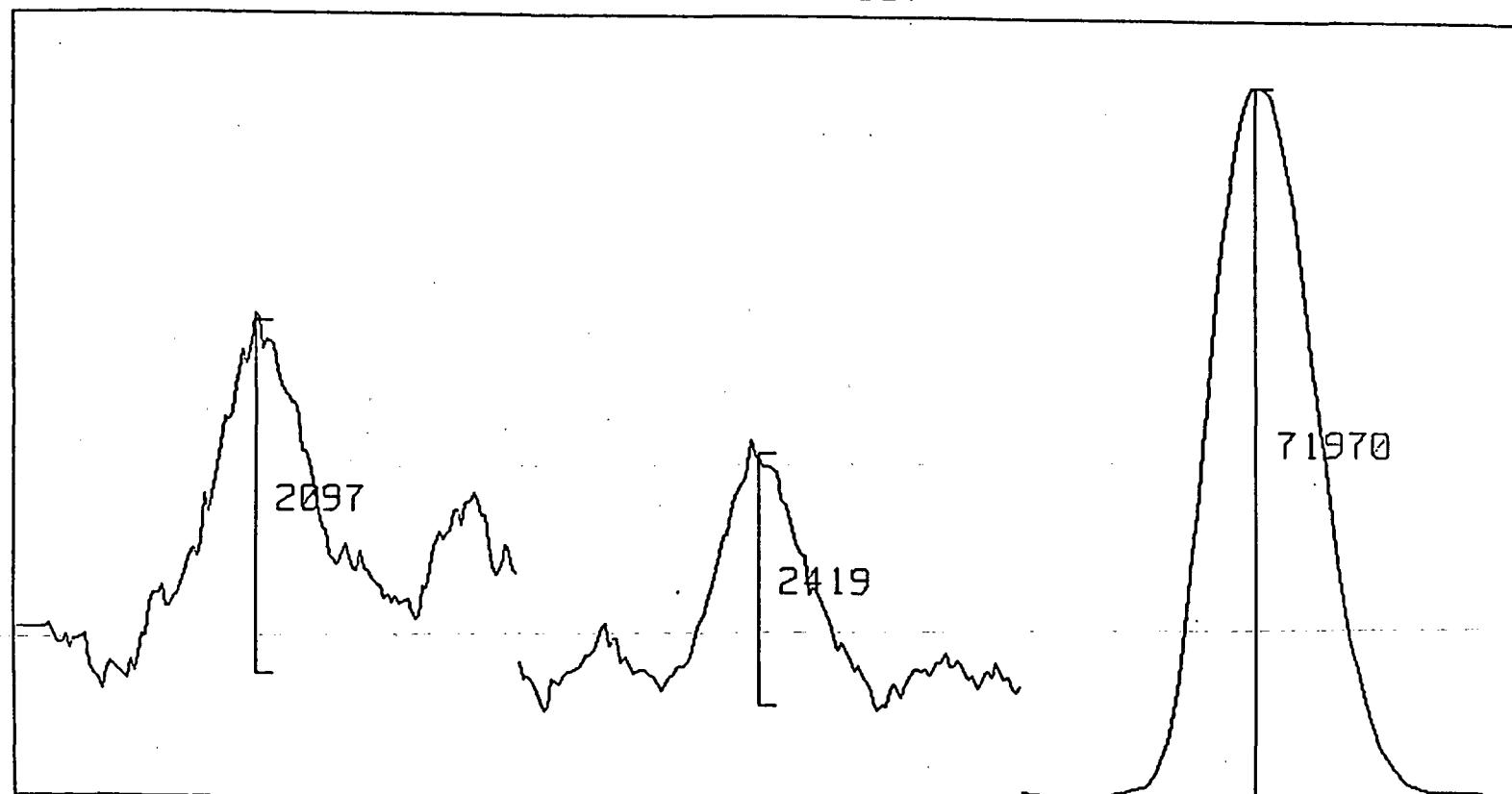
1815

MCMCS DIOXIN ANALYSIS      18:13:38  
RETN TIME: 8.24  
VOLUME RATIO: 5.00/ 39.00  
SYNTEX FILLET, AKJC4-015 SITE 4B

11/19/85      T10249.DX

Site 4B C Fillet +  
Remainder)

DET. LIMIT = 0.7 PPT  
% RECOVERY = 73.59%  
CONCENTRATION = 1.0 PPT  
RATIO = 0.867



|        |         |
|--------|---------|
| MASS   | 319.897 |
| GA IN  | 10.00   |
| 100%   | 466.1   |
| NO ISE | 1541.   |

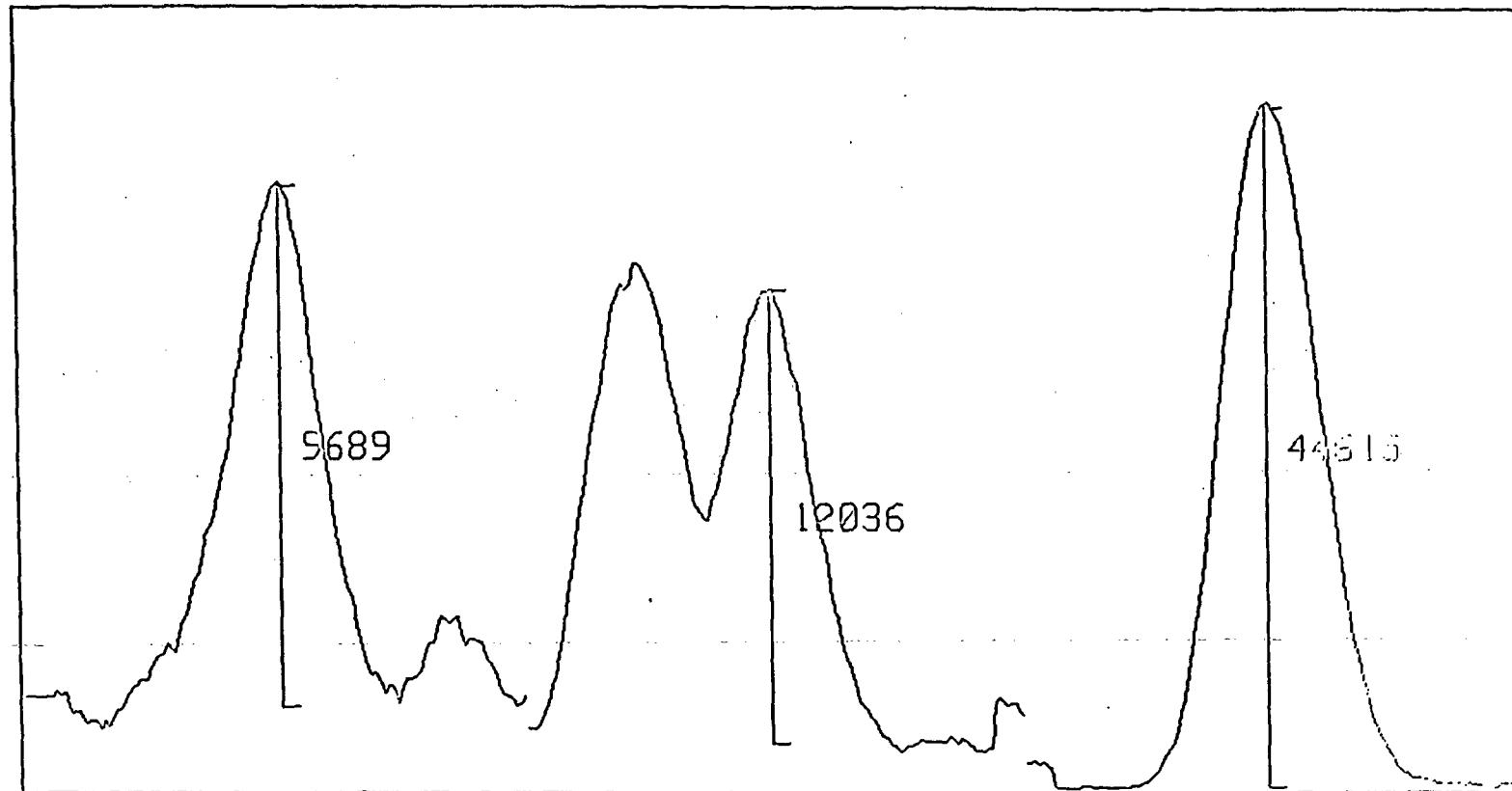
|        |         |
|--------|---------|
| MASS   | 321.894 |
| GA IN  | 10.00   |
| 100%   | 7560.   |
| NO ISE | 1806.   |

|        |         |
|--------|---------|
| MASS   | 333.934 |
| GA IN  | 4.00    |
| 100%   | 79437.  |
| NO ISE | 5532.   |

Site 4B (whole fish)

MCMS DIOXIN ANALYSIS    15:10:55    11/21/85    T10283.DX  
RETN TIME: 8.65  
VOLUME RATIO: 5.00 / 38.00    # SCANS: 14  
SYNTEX WHOLE FISH, AKJC4-016 SITE 4B

DET LIMIT = 2.1 PPT  
% RECOVERY = 50.33%  
CONCENTRATION = 6.2 PPT  
RATIO = 0.805

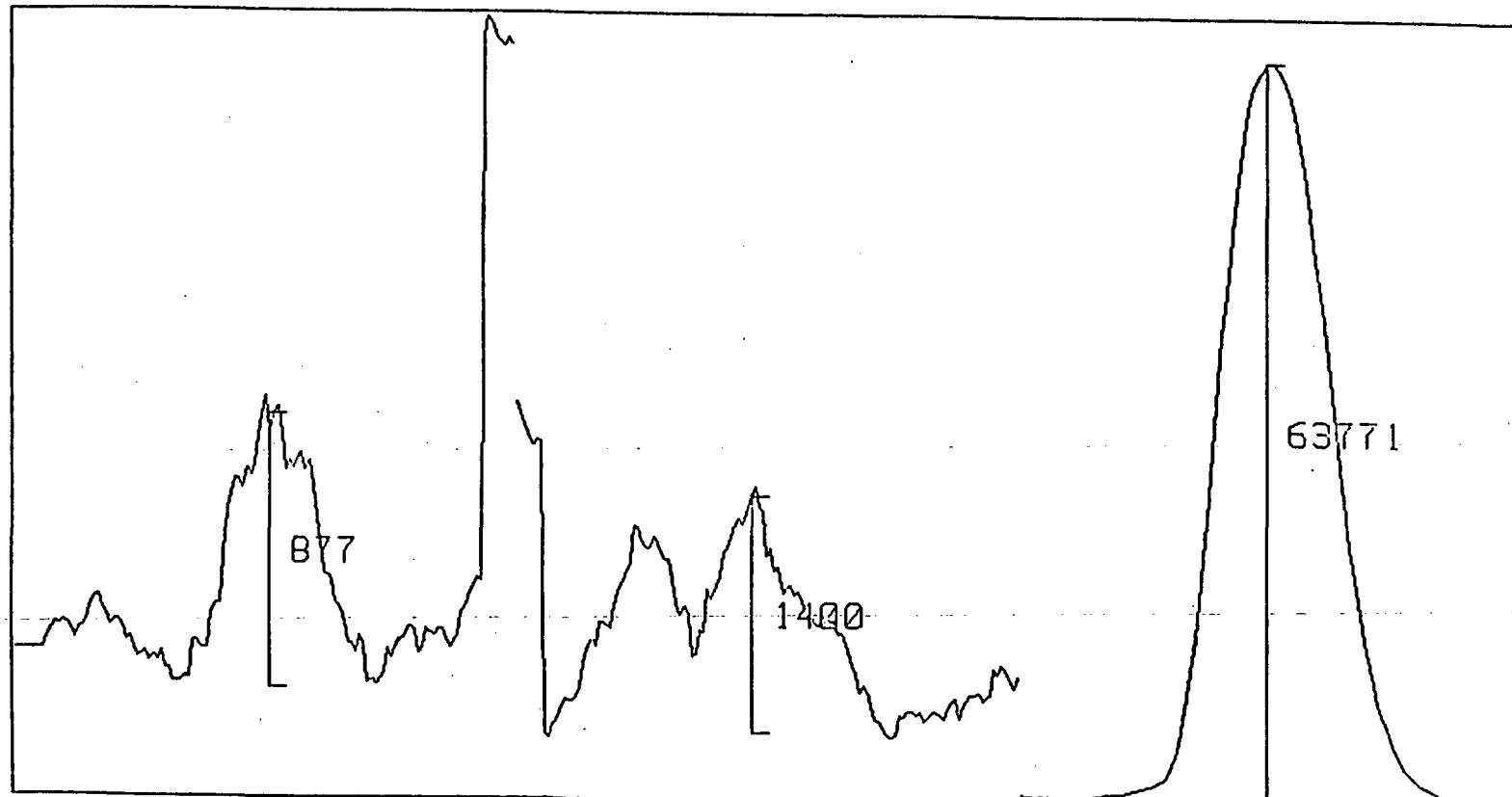


|        |         |         |         |
|--------|---------|---------|---------|
| MASS   | 319.897 | 321.894 | 333.994 |
| GA IN  | 10.00   | 10.00   | 4.00    |
| 100%   | 14636.  | 20823.  | 51518.  |
| NO ISE | 2866.   | 4171.   | 3495.   |

Site SA (Fillet)

MCMS DIOXIN ANALYSIS    21: 4:43    11/19/85    T10256.DX  
RETN TIME: 8.27  
VOLUME RATIO: 5.00 / 40.00    # SCANS: 15  
SYNTEX FILLET, AKJC4-01B SITE 5A

DET. LIMIT = 0.6 PPT  
% RECOVERY = 66.88%  
CONCENTRATION = 0.6 PPT  
RATIO = 0.626

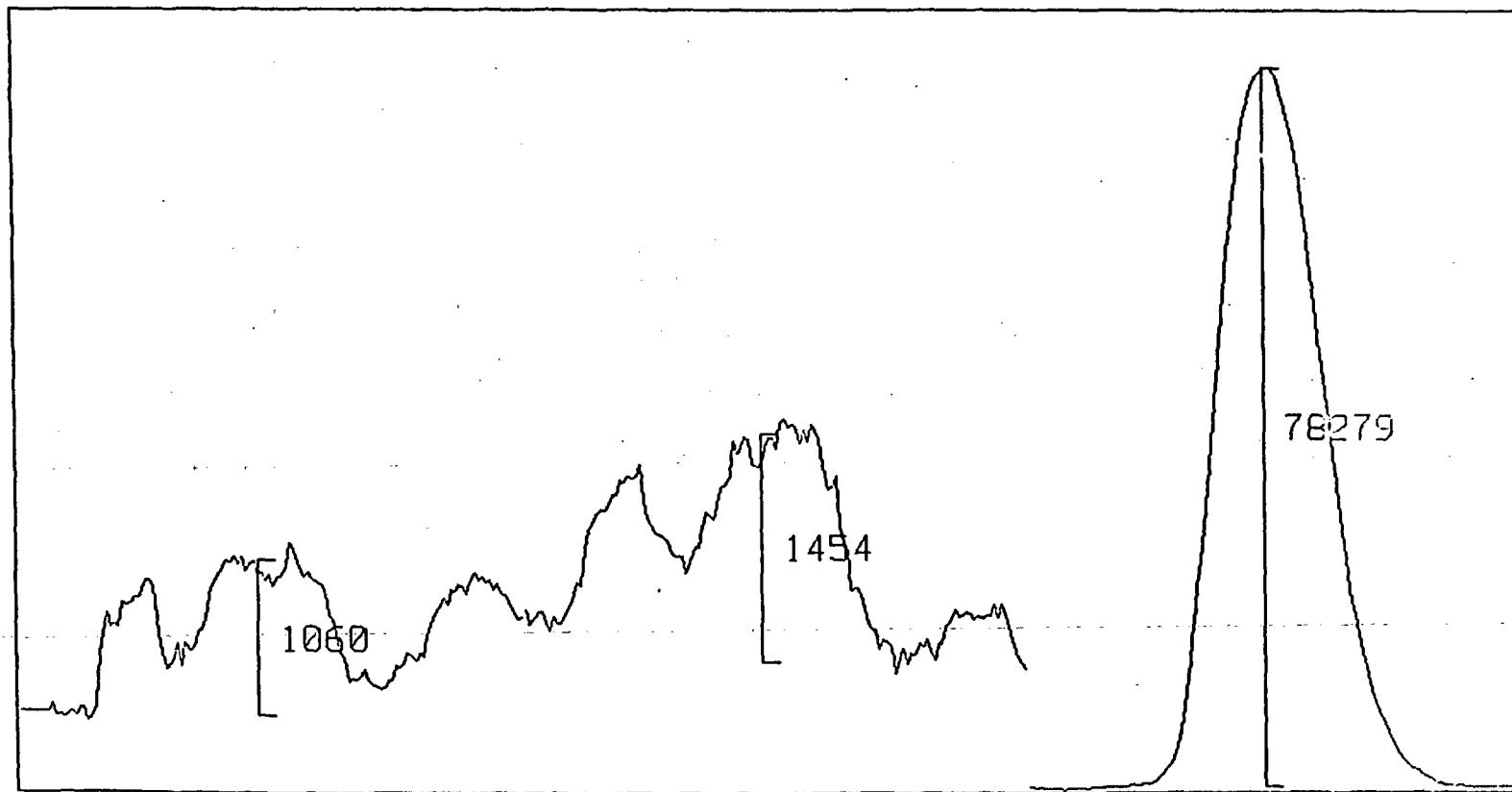


|        |         |         |         |
|--------|---------|---------|---------|
| MASS   | 319.897 | 321.894 | 333.934 |
| GA IN  | 10.00   | 10.00   | 4.00    |
| 100%   | 2520    | 4666    | 67986   |
| NO ISE | 894     | 1365    | 5510    |

Site 5B (Fillet +  
Remainder)

MCMS DIOXIN ANALYSIS    21:53: 4    11/19/85    T10257.DX  
RETN TIME: 8.26  
VOLUME RATIO: 5.00/ 38.00    # SCANS: 20  
SYNTEX FILLET, AKJC4-019 SITE 5B

DET. LIMIT = 0.5 PPT  
% RECOVERY = 77.99%  
CONCENTRATION = 0.5 PPT  
RATIO = 0.729

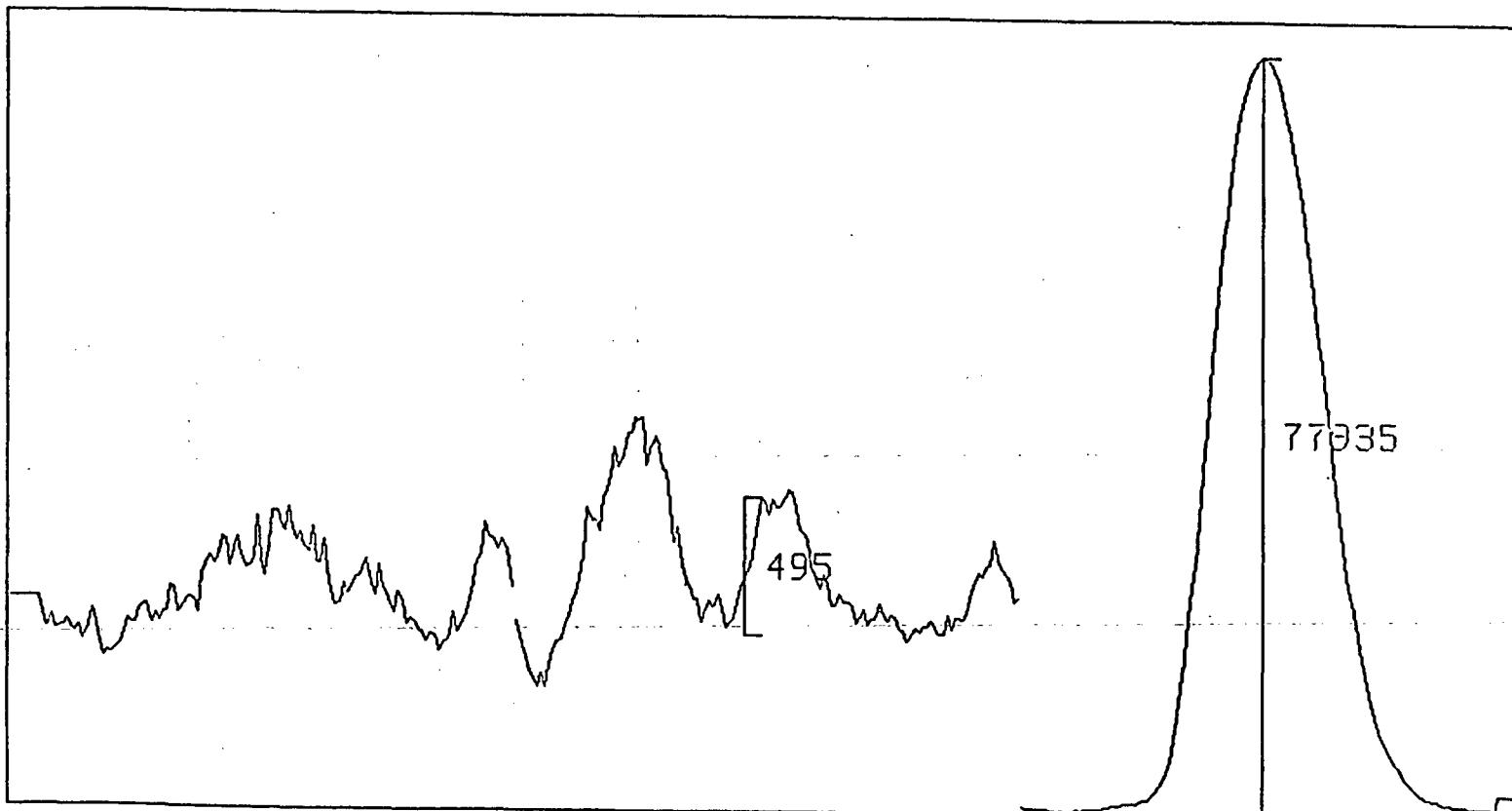


| MASS    | 319.897 | 321.894 | 333.934 |
|---------|---------|---------|---------|
| GA IN   | 10.00   | 10.00   | 4.00    |
| 100%    | 5355.   | 5014.   | 8527.   |
| NO [SE] | 1511.   | 1569.   | 7053.   |

Method Blank

MCMS DIOXIN ANALYSIS 19:47:29 11/19/85 T10253.DX  
RETN TIME: 8.28  
VOLUME RATIO: 5.00/ 42.00 # SCANS: 18  
BLANK FOR SYNTEX FILLET(11/11/85)

DET. LIMIT = 0.4 PPT  
% RECOVERY = 85.82%  
CONCENTRATION = 0.2 PPT

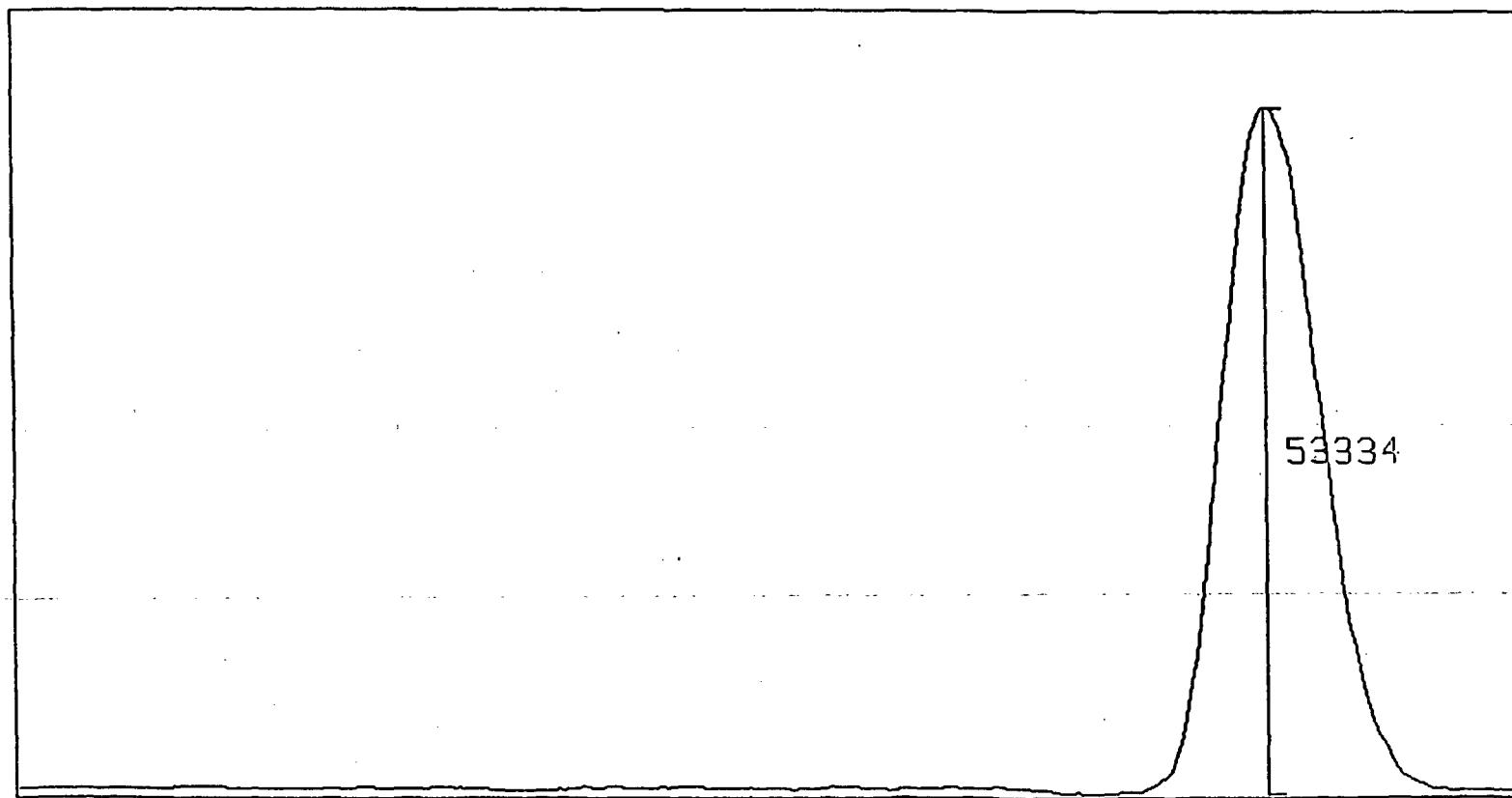


|       |         |         |         |
|-------|---------|---------|---------|
| MASS  | 319.897 | 321.894 | 333.934 |
| GA IN | 10.00   | 10.00   | 4.00    |
| 100%  | 2554.   | 2844.   | 81865.  |
| NOISE | 1063.   | 1028.   | 6047.   |

Method Blank:

MCMS DIOX IN ANALYSIS 16:35:54 11/19/85 T10247.DX  
RETN TIME: 8.47  
VOLUME RATIO: 5.00/ 48.00 # SCANS: 24  
BLANK FOR SYNTEX FILLET(11/8/85)

DET. LIMIT = 0.3 PPT  
% RECOVERY = 69.01%



|        |         |         |         |
|--------|---------|---------|---------|
| MASS   | 319.897 | 321.894 | 333.934 |
| GA IN  | 10.00   | 10.00   | 4.00    |
| 100%   | 61303.  | 61303.  | 61303.  |
| NO ISE | 556.    | 526.    | 4582.   |

SYNTEX RESEARCH  
ANALYTICAL RESEARCH  
PALO ALTO, CA 94304

RECEIVED  
JAN 07 1986

MEMORANDUM

SUPERFUND BRANCH

TO: Lewis Throop

A/R: 6662  
DATE: 10/31/85

CC: R. Forrester  
K. Stormer  
D. Robertson/B. Brunck/B. Berridge

FROM: D. Dei Rossi OOK

SUBJECT: Determination of 2,3,7,8-Tetrachlorodibenzo-p-Dioxin (2,3,7,8-TCDD) In Three Spring River Sediment Samples

The analysis for 2,3,7,8-TCDD in three Spring River Sediment Samples has been completed. Samples were analyzed using Syntex Research method #10,317B, "Determination of 2,3,7,8-Tetrachlorodibenzo-p-Dioxin (2,3,7,8-TCDD) in Soil, Sediment, and Sludge by Capillary Gas Chromatography Low Resolution Mass Spectrometry Selected Ion Monitoring (C-GC-LRMS-SIM)."

A summary of the analytical results is reported in Table I. Note that there was no 2,3,7,8-TCDD detected in any of the three samples at the reported detection limits. The computer analysis report and all mass chromatograms are attached.

Table I

| <u>Analytical Log No.</u> | <u>Sample I.D.</u> | <u>2,3,7,8-TCDD Content (PPT)</u> | <u>Comments</u>                |
|---------------------------|--------------------|-----------------------------------|--------------------------------|
| 85ENV 619                 | EPA #AKJC4023      | ND (3.1)                          | Loc. #1 0.3 miles downstream.  |
|                           |                    | ND (2.9)                          | Duplicate analysis             |
| 85ENV 620                 | EPA #AKJC4022      | ND (2.3)                          | Loc. #3 6.0 miles downstream.  |
| 85ENV 622                 | EPA #AKJC4021      | ND (2.5)                          | Loc. #5 12.0 miles downstream. |

ND = None Detected (Detection Limit in Parentheses)

PPT = Parts Per Trillion

Reference: TCDD9 pp. 1-27

Attachments

g/3srsssa.021

ANALYSIS REPORT 2.3.7.8-TCDD by C-GC-LRMS-SIM  
FILE:1147 Printed 10/01/85 17:19:02  
Compiled 09/20/85 10:44:50

| Log No.<br>(85ENV-)    | Sample<br>I.D. | Date<br>Sampled | Date<br>Extracted | Sample<br>Wt.(g) | 2,3,7,3-TCDD<br>Result (ppt) | -- Ion Ratios --<br>320/322 332/334 | Notes |
|------------------------|----------------|-----------------|-------------------|------------------|------------------------------|-------------------------------------|-------|
| STD. ADD.<br>STD. ADD. |                |                 |                   |                  |                              |                                     |       |
|                        |                | 9/13/85         |                   | 104.8            | 19.2                         | 0.73                                | 0.79  |
| 619                    | LOC. #1        | 8/27/85         | 9/13/85           | 97.0             | ND (3.1)                     | ---                                 | 0.78  |
| 620                    | LOC. #3        | 8/27/85         | 9/13/85           | 103.7            | ND (2.3)                     | ---                                 | 0.78  |
| 622                    | LOC. #3        | 8/27/85         | 9/13/85           | 101.4            | ND (2.5)                     | ---                                 | 0.79  |
| 619DUP                 | LOC. #1 DUP    | 8/27/85         | 9/16/85           | 98.0             | ND (2.9)                     | ---                                 | 0.81  |
| BLANK                  | BLANK          |                 | 9/16/85           | 100              | ND (1.2)                     | ---                                 | 0.79  |

NOTES :

- 1 ) Spring River Loc. #1 (0.3 miles downstream); EPA #AKJC4023
- 2 ) Spring River Loc. #2 (6.0 miles downstream); EPA #AKJC4022
- 3 ) Spring River Loc. #3 (12.0 miles downstream); EPA #AKJC4021
- 4 ) Standard addition (1.5795 ng TCDD) on blank soil 85ENV622 = 15.1 ppt
- 5 ) Blank sample - 100g Na<sub>2</sub>SO<sub>4</sub>
- 6 ) Duplicate analysis

===== RAW DATA =====

| Log No.<br>(85ENV-) | TCDD Spike (ng)<br>Native / 13C12 | SIM   | Area  | Response |        | Ion Ratio<br>(320+322)/(332+334) |
|---------------------|-----------------------------------|-------|-------|----------|--------|----------------------------------|
| Standard            | .3159 / 10                        | 0     | 10458 | 11680    | 453814 | 579737                           |
| Standard            | .6318 / 10                        | 0     | 8372  | 11700    | 185921 | 234966                           |
| Standard            | 1.264 / 10                        | 7608  | 28841 | 38874    | 323968 | 411510                           |
| Standard            | 3.159 / 10                        | 9758  | 43678 | 53941    | 185728 | 241402                           |
| STD. ADD.           | / 10                              | 16964 | 55591 | 76050    | 397299 | 504315                           |

| Log No.<br>(85ENV-) | TCDD Spike (ng)<br>Native / 13C12 | SIM (Peak Height(mm))s(Scale) |     |      |      | Ion Ratio -<br>(320+322)/(332+334) | RRE    |
|---------------------|-----------------------------------|-------------------------------|-----|------|------|------------------------------------|--------|
| Standard            | .3159 / 10                        | 174                           | 215 | 7814 | 9884 | 0.022                              | 0.6958 |
| Standard            | .6318 / 10                        | 138                           | 193 | 3170 | 3995 | 0.046                              | 0.7312 |

Mean RRE 0.7135

|        |      |         |         |       |       |        |
|--------|------|---------|---------|-------|-------|--------|
| 619    | / 10 | NL50    | NL52.2  | 5224  | 6683  | 0.0215 |
| 620    | / 10 | NL41.0  | NL50.0  | 5807  | 7447  | 0.0172 |
| 622    | / 10 | NL43.1  | NL59.8  | 6254  | 7953  | 0.0181 |
| 619DUP | / 10 | NL107.1 | NL162.7 | 14768 | 18318 | 0.0204 |
| BLANK  | / 10 | NL90.9  | NL77.6  | 22590 | 29553 | 0.0092 |

NL = Noise Level

ND = None Detected ( Detection Limit )

ppt = Parts Per Trillion

PL = Level of Interfering Peak

*Debra Johnson  
Rita A. Brink*

CALIBRATION REPORT: 2.3.7 TCDD by C-GC-THERM-SIM

Printed: 10/01/85

Compiled: 09/20/85

17:19:02 10:46:50

$S = \text{Ratio of Amounts Native TCDD (ng)/13C12-TCDD (ng)}$   
 $Y = \text{Ratio of SIM Response } (320+322)/(332+334)$

| Z       | Y      | Y(Reg.) | % Rel. Diff. (Y) |
|---------|--------|---------|------------------|
| 0.03159 | 0.0214 | 0.0231  | -7.74            |
| 0.06318 | 0.0477 | 0.0466  | 3.61             |
| 0.1264  | 0.0921 | 0.0917  | 0.36             |
| 0.3155  | 0.2285 | 0.2298  | -0.13            |

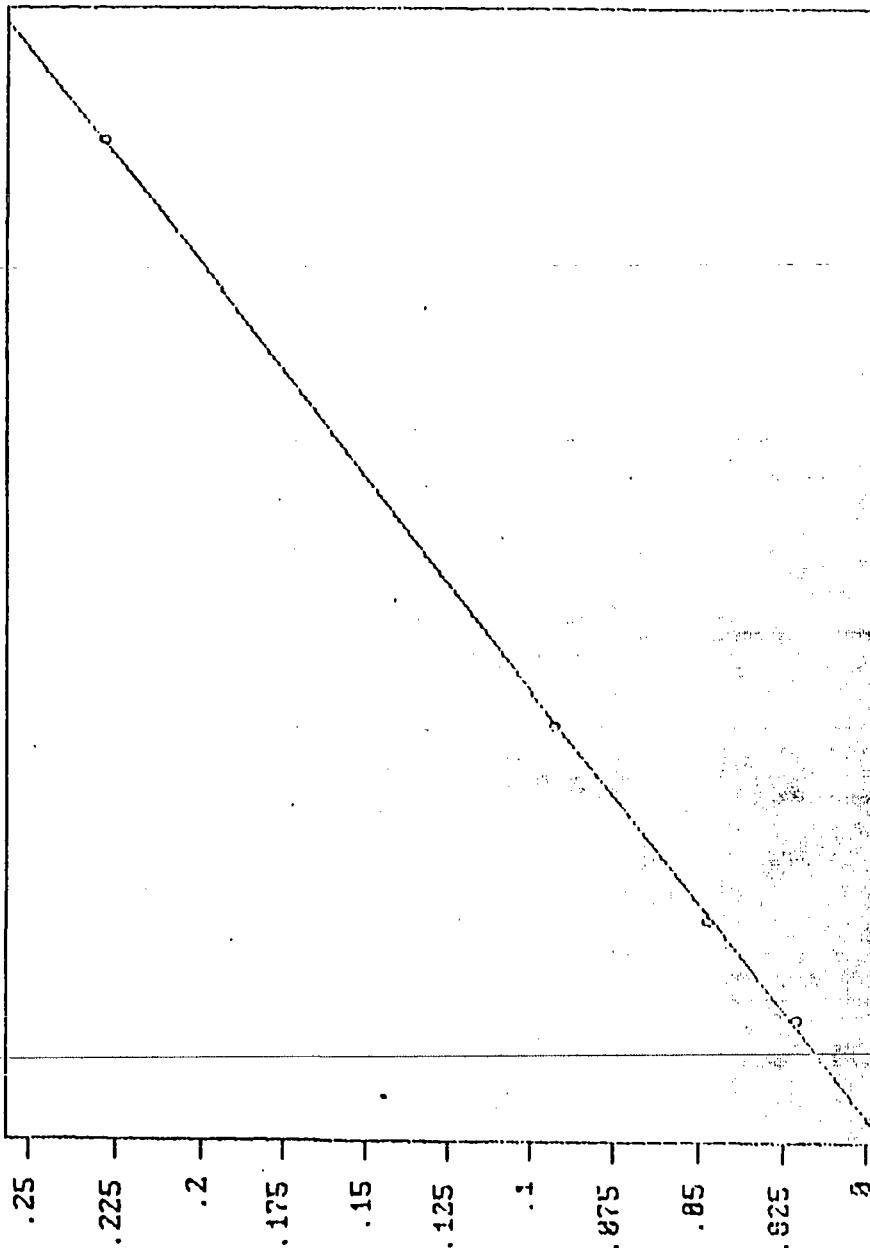
$$Y = 0.72351(Z) + 0.00029$$

Correlation Coefficient: 0.9998817

Standard Error of Estimate: 0.0017362

GPT?

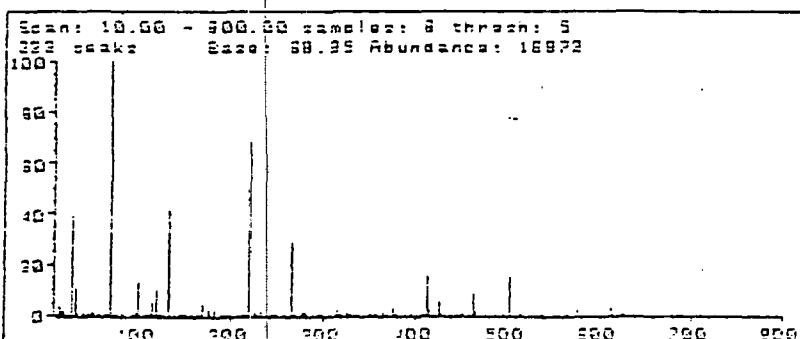
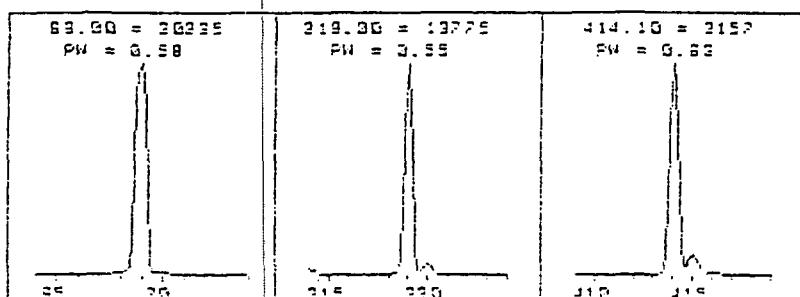
Ratio of SIM Response  $(320+322)/(332+334)$



Ratio of SIM Response Native TCDD (ng)/13C12-TCDD (ng)  
= 0.72351 Z + 0.00029

MANUAL TUNE ATU 0 23 Sep 85 10:05 am

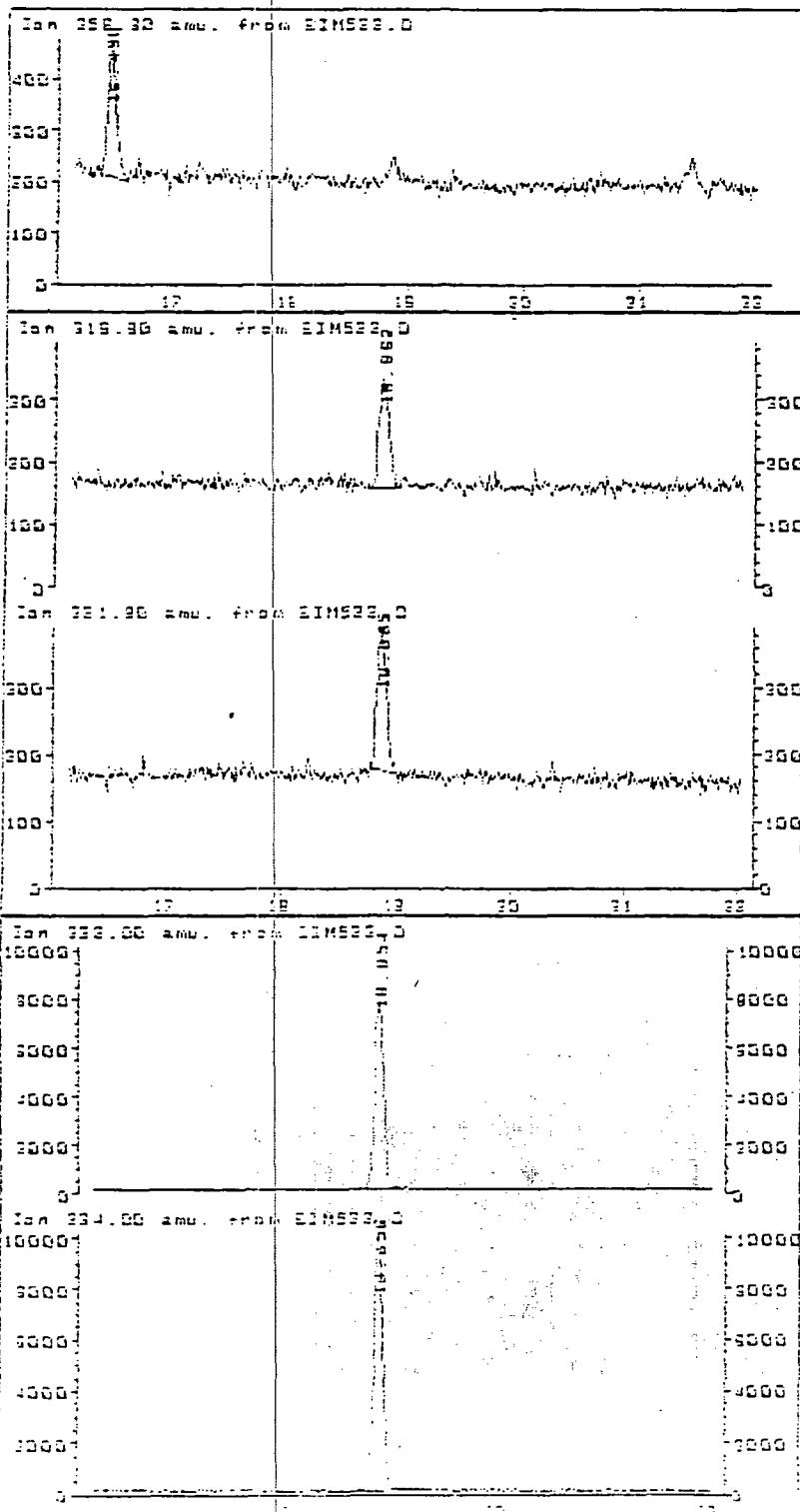
9 Multicollect 2200  
AMU gain 136 AMU offset 52 Emission ON  
Ion focus 0.0 Entr. lens 45 Repeller 10.20  
Axis gain 15 X-ray 00.0  
Samples 4 Axis offset 10  
Averages 1 Stabilize 0.10



| Mass   | Abund | Rel Abund | Iso Mass | Iso Abund | Iso Ratio |
|--------|-------|-----------|----------|-----------|-----------|
| 68.35  | 16872 | 100.00    | 68.85    | 191       | 1.13      |
| 219.00 | 11429 | 67.74     | 220.00   | 198       | 4.36      |
| 414.05 | 2599  | 15.40     | 414.55   | 360       | 10.30     |

Operator: D. DEI ROSSI      Date: 23 Sep 95 10:32 am  
 Sample Info : STANDARO 10.1  
 Miss Info : 0.3159 ng TCD0  
 Integration File Name : SIMS22.I  
 =====  
 Report by Retention Time (min)

STD 10-1



水木天

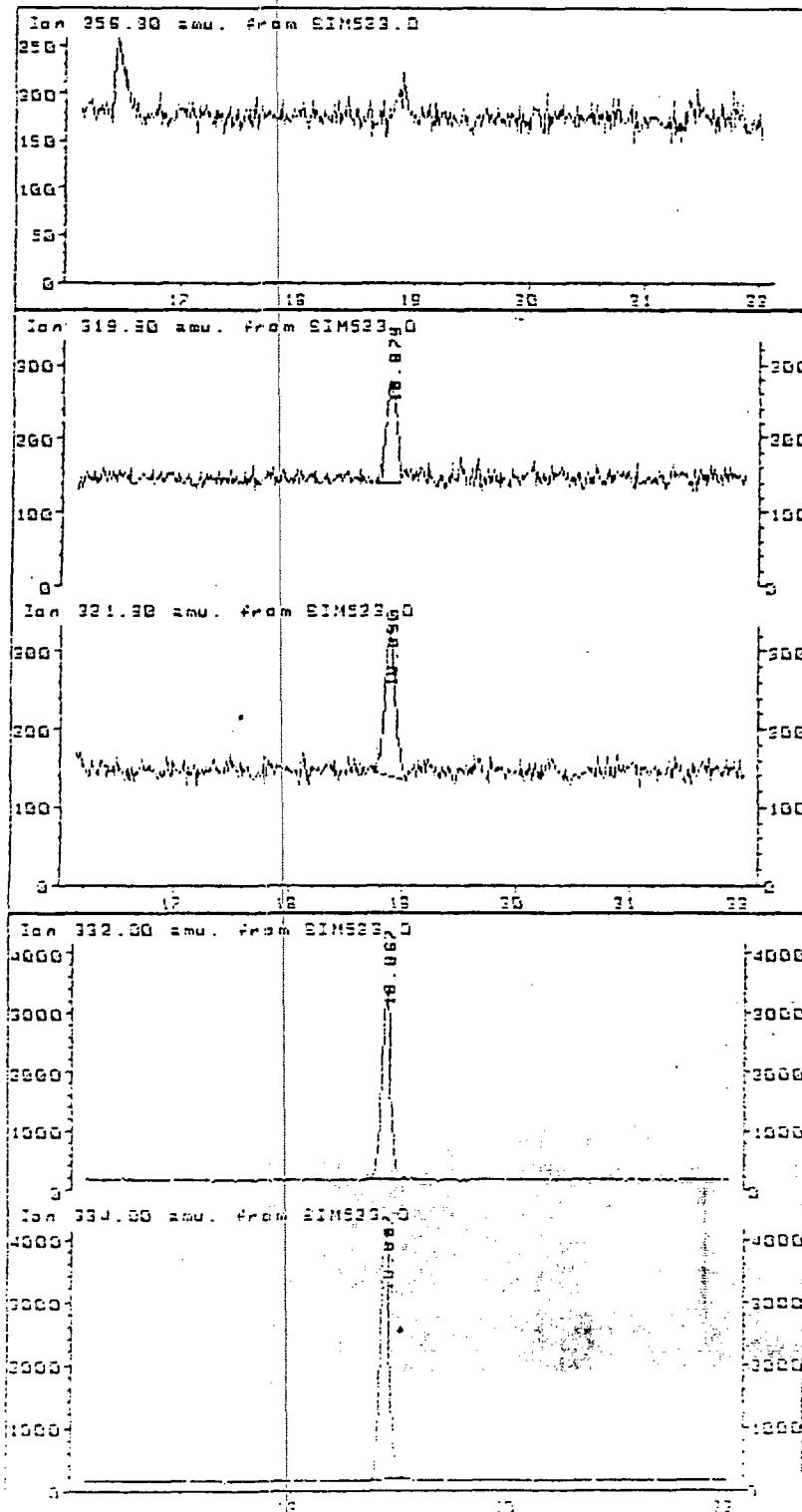
Operator: D. DEI ROSSI  
Sample Info : STANDARD 10.2  
Misc Info: 0.6315 mg TCDD  
Integration File Name : SIMS23.1

卷之三

## Report by Retention Time

| Ret Time | Signal | Descr  | Type | Area | Height  | % PK | % SG  | % Lek  | % Ldg  |
|----------|--------|--------|------|------|---------|------|-------|--------|--------|
| 18.367   | Mass   | 354.90 | amu  | 9.9  | 234.956 | 3695 | 55.29 | 100.00 | 100.00 |
| 18.367   | Mass   | 352.90 | amu  | 9.9  | 185.321 | 3170 | 42.16 | 100.00 | 79.15  |
| 18.390   | Mass   | 321.90 | amu  | 8.9  | 117.00  | 153  | 2.65  | 100.00 | 4.88   |
| 18.379   | Mass   | 319.90 | amu  | 8.9  | 93.72   | 132  | 1.90  | 100.00 | 3.56   |

STD 10-2



卷之三

Operator: D. DEI ROSSI  
Sample Info : STANDARD 10.3  
Misc Info: 1.254 mg T000  
Integration File Name : SIM524.1

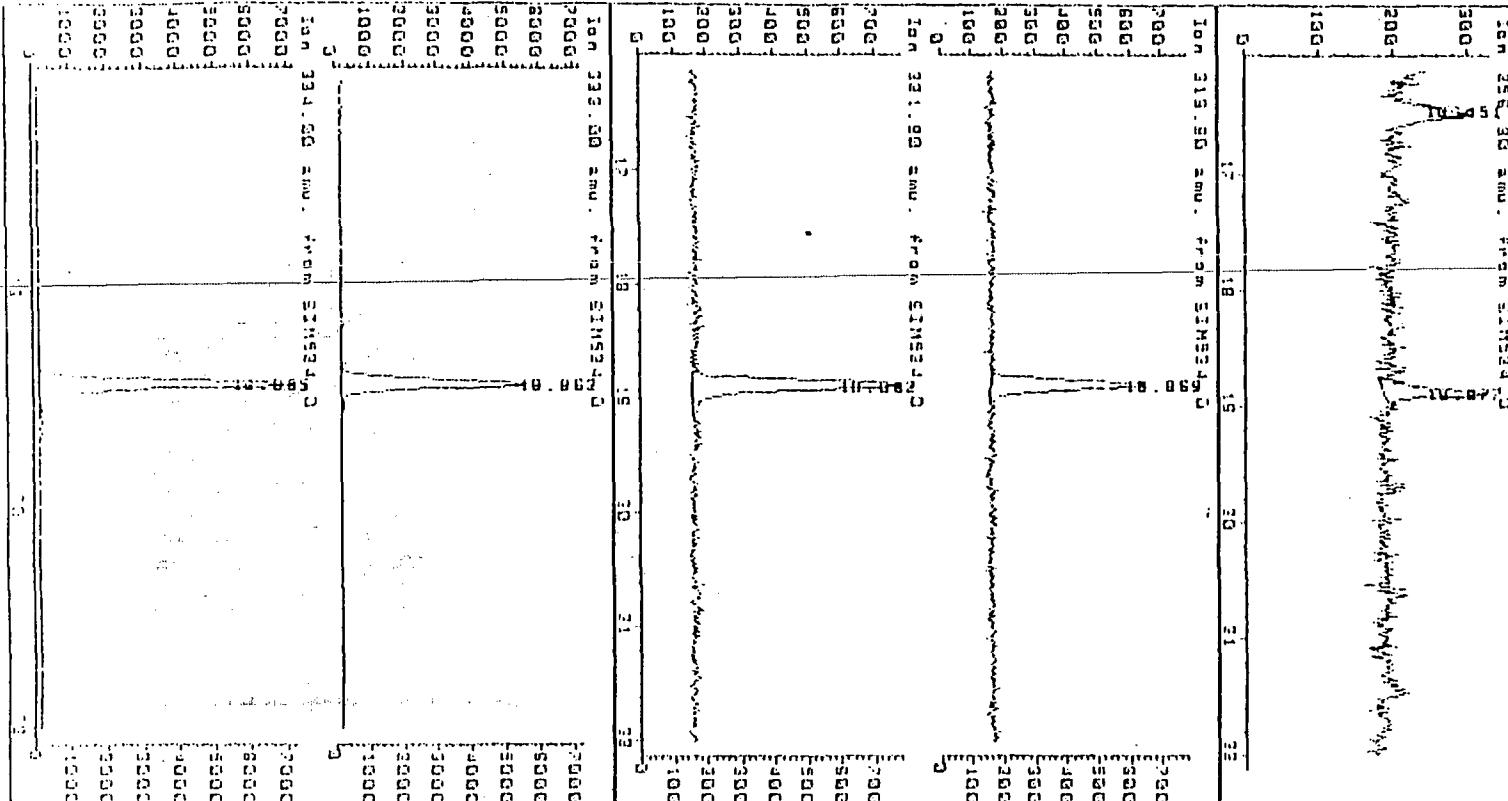
23 Sep 95 11:49 am

Report by Retention Time

Operator: D. DEI ROSSI  
Sample Info : STANDARD 10.3  
MS Info: 1.264 mg TCD0  
Integration File Name : SIM524.I

| Ret Time | Signal Descr | Type          | Area   | Height | % Pk   | % Sg   | % LPK  | % LSG  |
|----------|--------------|---------------|--------|--------|--------|--------|--------|--------|
| 16.451   | Mass         | 256.90 amu PP | 108.1  | 40     | 100.00 | 12.54  | 100.00 | 14.34  |
| 16.835   | Mass         | 254.90 amu SP | 4115.0 | 7115   | 50.75  | 100.00 | 100.00 | 100.00 |
| 16.852   | Mass         | 332.90 amu PP | 3239.6 | 552.0  | 38.36  | 100.00 | 78.73  | 120.00 |
| 16.862   | Mass         | 321.90 amu UP | -      | 3867.4 | 626    | 4.79   | 100.00 | 9.45   |
| 16.866   | Mass         | 319.90 amu SP | 2994.1 | 499    | 3.56   | 100.00 | 7.01   | 100.00 |
| 16.974   | Mass         | 255.90 amu PP | 750.6  | 140    | 0.94   | 87.46  | 1.35   | 100.00 |

10.3



## \*\*\* Area Percent \*\*\*

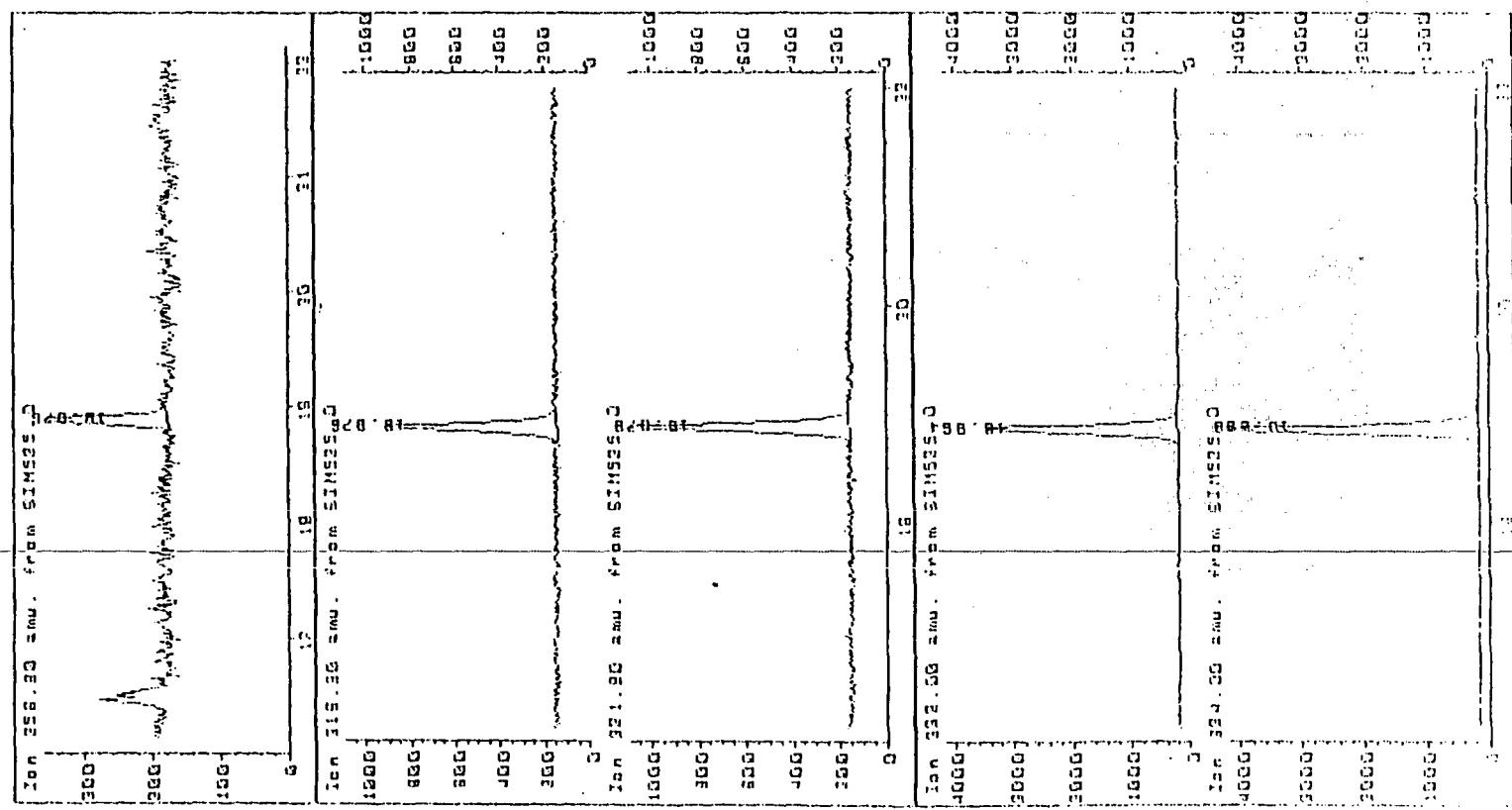
===== Report by Retention Time =====

Operator: D. DEI ROSSI  
Sample Info : STANDARD 10.4  
Misc Info: 3.153 ng TCDD  
Integration File Name : SIM525.I

23 Sep 95 12:44 pm

| Ret Time | Signal Descr | Type            | Area   | Height | % Pt  | % Sq   | % LPk  | % LSq  |
|----------|--------------|-----------------|--------|--------|-------|--------|--------|--------|
| 18.862   | Mass         | 334.00 amu BP   | 241402 | 4022   | 45.15 | 100.00 | 100.00 | 100.00 |
| 18.864   | Mass         | 332.00 amu BP   | 185728 | 3244   | 34.75 | 100.00 | 76.94  | 100.00 |
| 18.878   | Mass         | 321.90 amu PP   | 53841  | 937    | 10.09 | 100.00 | 22.34  | 100.00 |
| 18.876   | Mass         | 319.90 amu VP   | 43678  | 758    | 3.17  | 100.00 | 18.09  | 100.00 |
| 18.876   | Mass         | 255.90 amu PP " | 8752   | 182    | 1.63  | 100.00 | 4.04   | 100.00 |

STD 10-4



\*\*\* Area Percent \*\*\*

Operator: O. DEI ROSSI

Sample Info : BLANK

Misc Info:

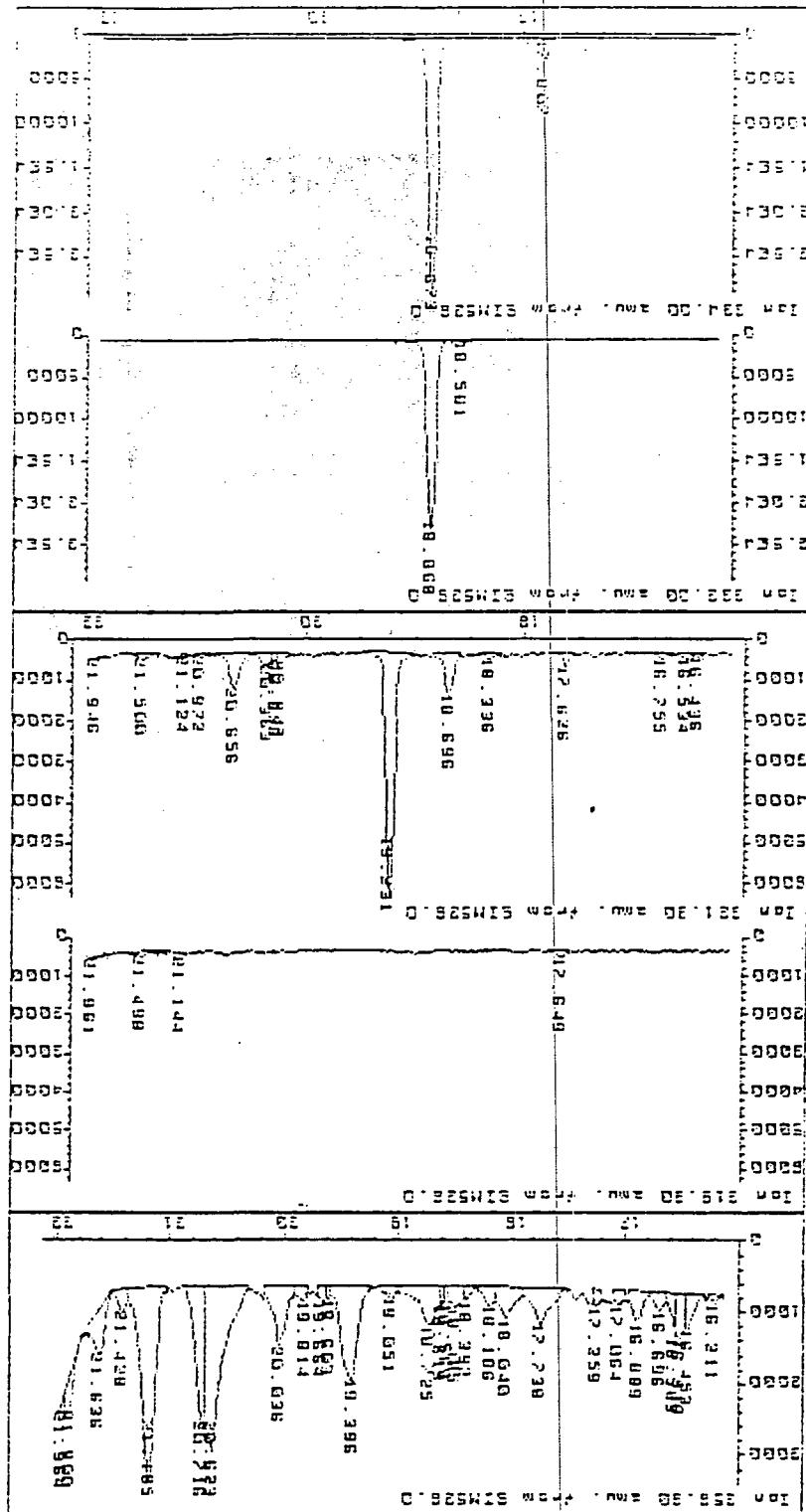
Integration File Name : SIM525.I

Report By Retention Time

23 Sep 25 1:21 PM

| Ret Time | Signal Descr | Type          | Area  | Height | % Fw   | % Se  | % Lfw  | % Lse |
|----------|--------------|---------------|-------|--------|--------|-------|--------|-------|
| 16.211   | Mass         | 256.90 amu UV | 3924  | 126    | 100.00 | 0.32  | 100.00 | 1.39  |
| 16.436   | Mass         | 321.90 amu PU | 2990  | 110    | 100.00 | 0.55  | 100.00 | 0.82  |
| 16.453   | Mass         | 256.90 amu UV | 23968 | 524    | 100.00 | 1.97  | 100.00 | 10.27 |
| 16.518   | Mass         | 256.90 amu UV | 23562 | 651    | 100.00 | 2.52  | 100.00 | 12.15 |
| 16.534   | Mass         | 321.90 amu UV | 8365  | 158    | 100.00 | 1.49  | 100.00 | 2.56  |
| 16.563   | Mass         | 256.90 amu UV | 20087 | 523    | 100.00 | 1.65  | 100.00 | 5.61  |
| 16.636   | Mass         | 256.90 amu UV | 13857 | 272    | 100.00 | 1.14  | 100.00 | 5.84  |
| 16.755   | Mass         | 321.90 amu UP | 6729  | 146    | 100.00 | 1.26  | 100.00 | 2.07  |
| 16.889   | Mass         | 256.90 amu UV | 25974 | 455    | 100.00 | 2.15  | 100.00 | 11.15 |
| 17.064   | Mass         | 256.90 amu UV | 9404  | 222    | 100.00 | 0.77  | 100.00 | 4.02  |
| 17.259   | Mass         | 256.90 amu UV | 2713  | 268    | 100.00 | 0.71  | 100.00 | 2.72  |
| 17.626   | Mass         | 321.90 amu UV | 17741 | 354    | 100.00 | 3.16  | 100.00 | 5.46  |
| 17.649   | Mass         | 319.20 amu UP | 10712 | 191    | 100.00 | 26.98 | 100.00 | 75.20 |
| 17.736   | Mass         | 256.90 amu PU | 58566 | 511    | 100.00 | 4.79  | 100.00 | 5.31  |
| 17.397   | Mass         | 254.20 amu UP | 6362  | 121    | 100.00 | 0.41  | 100.00 | 0.42  |
| 18.040   | Mass         | 256.90 amu UV | 41912 | 453    | 100.00 | 5.44  | 100.00 | 17.35 |
| 18.156   | Mass         | 256.90 amu UV | 16528 | 274    | 100.00 | 1.36  | 100.00 | 7.02  |
| 18.326   | Mass         | 321.90 amu UV | 11524 | 172    | 100.00 | 2.11  | 100.00 | 5.55  |
| 18.380   | Mass         | 256.90 amu UV | 5995  | 128    | 100.00 | 0.57  | 100.00 | 0.52  |
| 18.435   | Mass         | 256.20 amu UP | 12154 | 230    | 100.00 | 1.31  | 100.00 | 7.51  |
| 18.551   | Mass         | 256.90 amu UV | 1259  | 125    | 100.00 | 1.06  | 100.00 | 1.16  |
| 18.553   | Mass         | 256.90 amu UV | 1251  | 125    | 100.00 | 1.06  | 100.00 | 1.16  |
| 18.555   | Mass         | 256.90 amu UV | 1253  | 125    | 100.00 | 1.06  | 100.00 | 1.16  |

|        |      |        |     |          |          |       |        |        |        |
|--------|------|--------|-----|----------|----------|-------|--------|--------|--------|
| 18.373 | Mass | 334.39 | PP  | 16789.09 | 26553    | 55.30 | 19.53  | 100.00 | 120.00 |
| 19.352 | Mass | 332.50 | amu | PP       | 15298.57 | 25590 | 44.20  | 95.95  | 79.23  |
| 19.051 | Mass | 253.35 | amu | UV       | 11255    | 167   | 100.00 | 0.32   | 100.00 |
| 19.231 | Mass | 321.36 | amu | UV       | 324835   | 6305  | 100.00 | 57.27  | 130.00 |
| 19.396 | Mass | 255.36 | amu | UV       | 152979   | 1307  | 100.00 | 10.90  | 100.00 |
| 19.603 | Mass | 256.30 | amu | UV       | 5535     | 203   | 100.00 | 0.29   | 100.00 |
| 19.664 | Mass | 256.35 | amu | UV       | 8285     | 226   | 100.00 | 0.52   | 100.00 |
| 19.314 | Mass | 253.36 | amu | UV       | 11995    | 212   | 100.00 | 0.99   | 100.00 |
| 20.035 | Mass | 258.36 | amu | UV       | 57078    | 659   | 100.00 | 5.50   | 100.00 |
| 20.247 | Mass | 321.36 | amu | UV       | 10497    | 258   | 100.00 | 1.67   | 100.00 |
| 20.276 | Mass | 321.36 | amu | UV       | 8637     | 256   | 100.00 | 1.54   | 100.00 |
| 20.363 | Mass | 321.36 | amu | UV       | 20625    | 274   | 100.00 | 3.55   | 100.00 |
| 20.523 | Mass | 253.36 | amu | UV       | 233364   | 2313  | 100.00 | 19.14  | 100.00 |
| 20.656 | Mass | 321.36 | amu | UV       | 53741    | 695   | 100.00 | 11.35  | 100.00 |
| 20.716 | Mass | 255.36 | amu | UV       | 106141   | 1983  | 100.00 | 8.70   | 100.00 |
| 20.972 | Mass | 321.36 | amu | UV       | 8190     | 111   | 100.00 | 1.45   | 100.00 |
| 21.124 | Mass | 321.36 | amu | UV       | 14293    | 206   | 100.00 | 39.37  | 100.00 |
| 21.144 | Mass | 319.36 | amu | UV       | 215448   | 2712  | 100.00 | 17.57  | 100.00 |
| 21.428 | Mass | 255.36 | amu | UV       | 15207    | 223   | 100.00 | 1.25   | 100.00 |
| 21.500 | Mass | 321.36 | amu | PP       | 10695    | 142   | 55.36  | 1.94   | 100.00 |
| 21.498 | Mass | 319.36 | amu | PP       | 6776     | 117   | 44.81  | 24.56  | 90.54  |
| 21.536 | Mass | 256.36 | amu | PP       | 46251    | 836   | 100.00 | 5.79   | 100.00 |
| 21.900 | Mass | 256.36 | amu | UV       | 16525    | 452   | 100.00 | 1.50   | 100.00 |
| 21.946 | Mass | 321.36 | amu | PP       | 413      | 32    | 100.00 | 0.37   | 100.00 |
| 21.361 | Mass | 319.36 | amu | PP       | 1988     | 59    | 21.28  | 5.50   | 15.52  |
| 21.354 | Mass | 256.36 | amu | UV       | 1516     | 197   | 33.72  | 0.35   | 100.00 |



BANK

BLANK

בְּרִית מָשֶׁה - כְּלֵי קָדְשָׁה - בְּרִית מָשֶׁה - כְּלֵי קָדְשָׁה

卷之三

22,590

## \*\*\* Area Percent \*\*\*

=====  
==== Report by Retention Time =====  
=====

Operator: D. DEI ROSSI

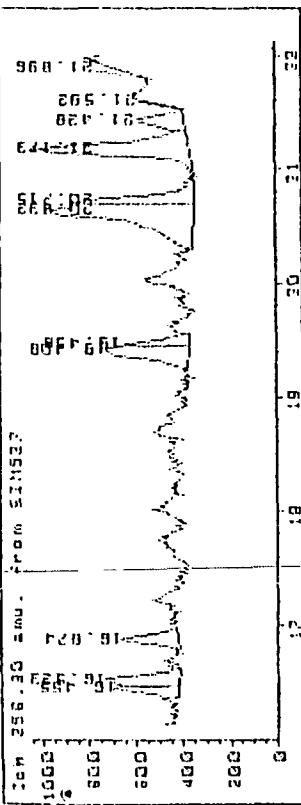
23 Sep 85 2:23 pm

Sample Info : 85ENVE19

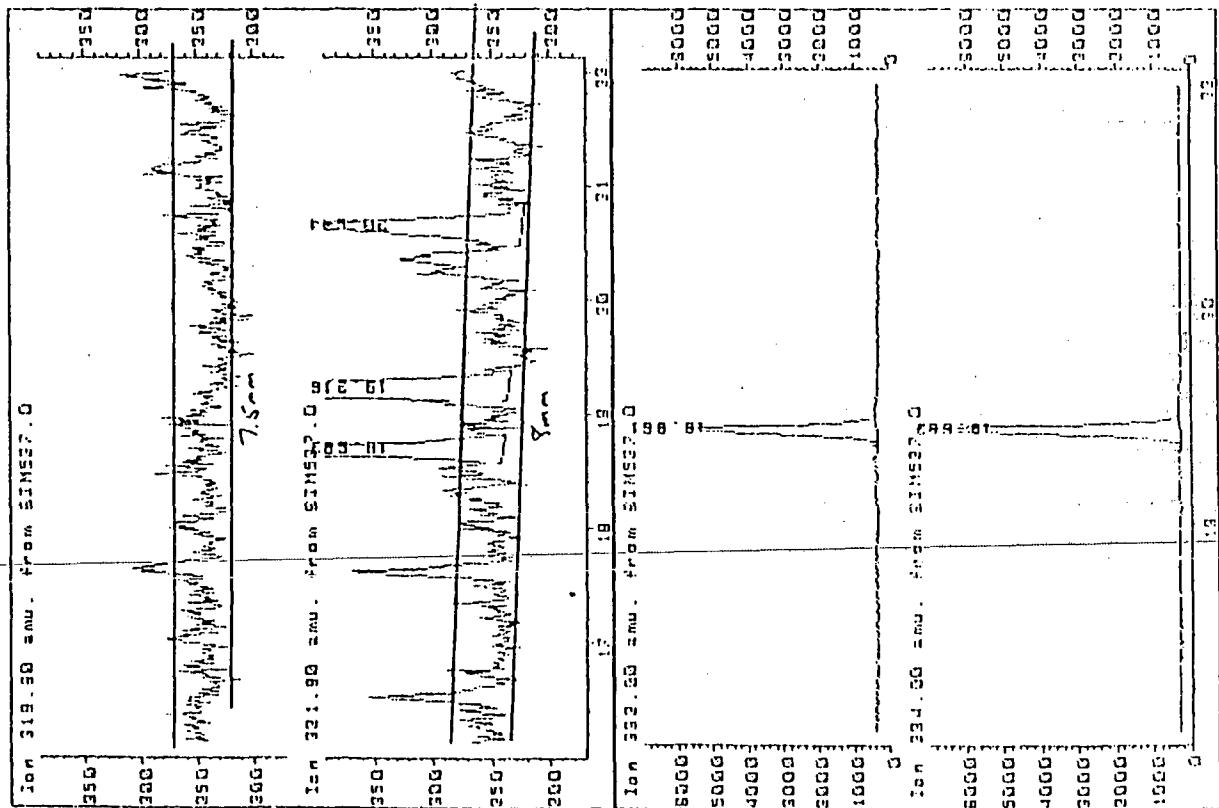
Misc Info: SEDIMENT SAMPLE LOC. #1, 57.0 g sample

Integration File Name : SIME27.I

| Ret Time | Signal Descr | Type          | Area   | Height | % PK   | % Sq   | % LPk  | % Lsq  |
|----------|--------------|---------------|--------|--------|--------|--------|--------|--------|
| 16.455   | Mass         | 256.90 amu UV | 10642  | 276    | 100.00 | 3.57   | 100.00 | 15.45  |
| 16.519   | Mass         | 321.90 amu VP | 6493   | 119    | 25.90  | 3.77   | 34.85  | 5.82   |
| 16.523   | Mass         | 256.90 amu UV | 18534  | 322    | 74.10  | 6.23   | 100.00 | 27.00  |
| 16.874   | Mass         | 256.90 amu PV | 13933  | 297    | 100.00 | 4.97   | 100.00 | 20.23  |
| 17.227   | Mass         | 256.90 amu VV | 6357   | 131    | 100.00 | 2.13   | 100.00 | 9.23   |
| 17.627   | Mass         | 321.90 amu VP | 6209   | 135    | 100.00 | 3.95   | 100.00 | 5.89   |
| 17.743   | Mass         | 256.90 amu UV | 7603   | 103    | 100.00 | 2.55   | 100.00 | 11.04  |
| 18.002   | Mass         | 256.90 amu PV | 10097  | 135    | 100.00 | 3.38   | 100.00 | 14.65  |
| 18.863   | Mass         | 321.90 amu UV | 32299  | 422    | 100.00 | 12.95  | 100.00 | 19.30  |
| 18.889   | Mass         | 256.90 amu PP | 5130   | 105    | 100.00 | 1.72   | 100.00 | 7.45   |
| X 18.962 | Mass         | 324.00 amu PP | 384431 | 6883   | 55.70  | 100.00 | 100.00 | 100.00 |
| X 18.961 | Mass         | 332.00 amu PP | 306735 | 5224   | 44.30  | 100.00 | 78.52  | 100.00 |
| 18.216   | Mass         | 321.90 amu VV | 115529 | 2333   | 100.00 | 87.07  | 100.00 | 100.00 |
| 18.408   | Mass         | 256.90 amu PV | 23614  | 353    | 100.00 | 7.81   | 100.00 | 34.29  |
| 18.458   | Mass         | 256.90 amu VV | 11166  | 301    | 100.00 | 3.74   | 100.00 | 16.21  |
| 20.033   | Mass         | 256.90 amu UV | 14736  | 121    | 100.00 | 4.31   | 100.00 | 21.40  |
| 20.644   | Mass         | 321.90 amu VP | 21121  | 298    | 13.47  | 10.26  | 10.87  | 18.26  |
| 20.632   | Mass         | 256.90 amu UV | 66870  | 630    | 76.53  | 22.07  | 100.00 | 100.00 |
| 20.715   | Mass         | 256.90 amu VP | 22242  | 522    | 100.00 | 7.45   | 100.00 | 32.30  |
| 21.173   | Mass         | 256.90 amu PV | 50665  | 537    | 100.00 | 15.92  | 100.00 | 73.59  |
| 21.426   | Mass         | 256.90 amu VP | 12767  | 226    | 100.00 | 4.28   | 100.00 | 16.84  |
| 21.380   | Mass         | 256.90 amu PV | 2842   | 130    | 100.00 | 1.29   | 100.00 | 5.32   |
| 21.378   | Mass         | 256.90 amu P  | 10710  | 183    | 100.00 | 0.48   | 100.00 | 11.37  |
| 21.388   | Mass         | 256.90 amu PV | 7176   | 176    | 100.00 | 1.73   | 100.00 | 11.37  |



85ENV619



522.4

524.5

\*\*\* Area Percent \*\*\*

Operator: D. DEI ROSSI

Sample Info : BSENV020

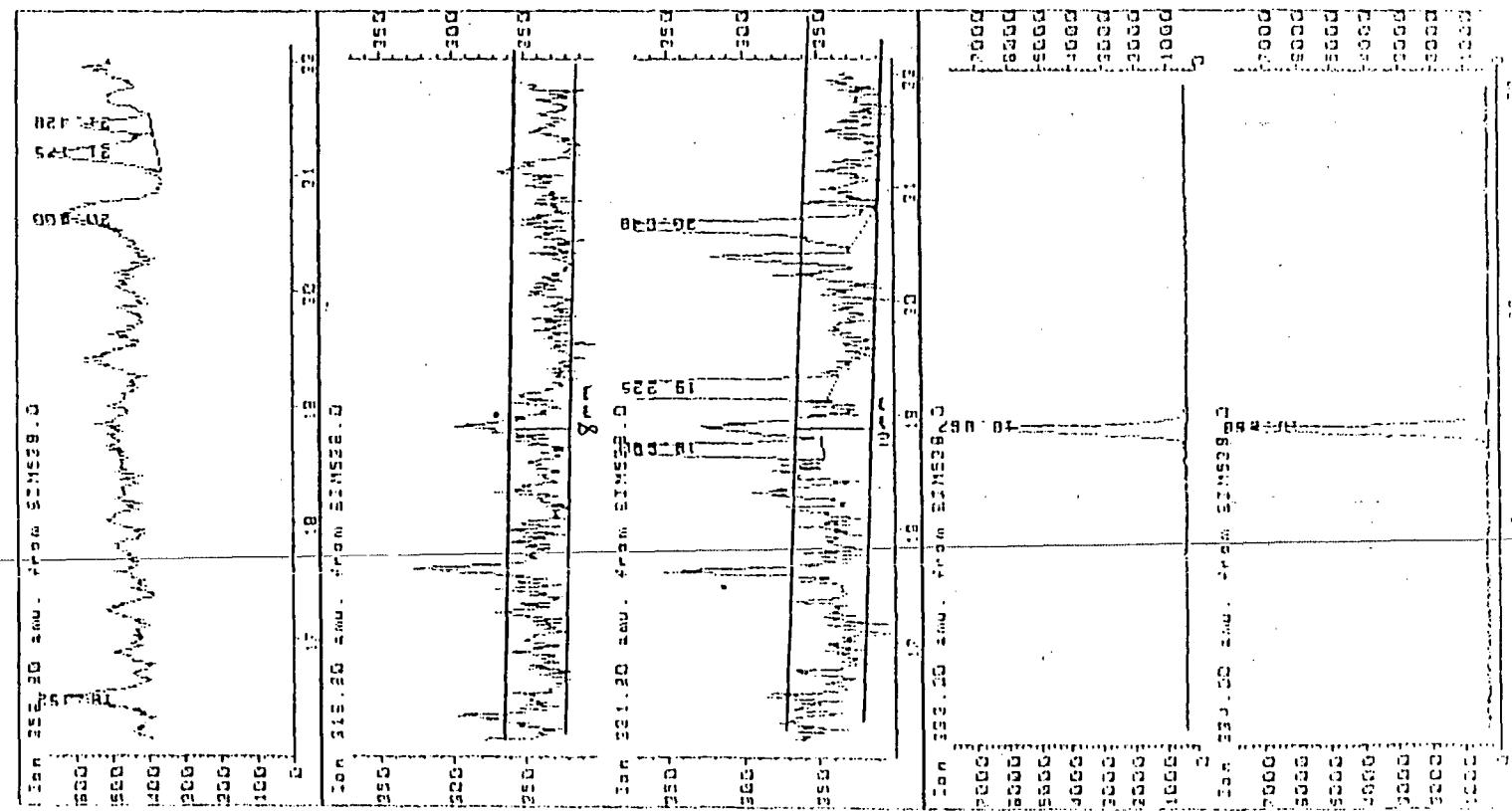
Misc Info: SEDIMENT SAMPLE LOC. #3, 103.7g sample

Integration File Name : SIME2E.I

22 Sep 22 3:11 pm

| Ret Time | Signal Descr | Type          | Area   | Height | % PK   | % 59   | % Lnk  | % Leg  |
|----------|--------------|---------------|--------|--------|--------|--------|--------|--------|
| 16.452   | Mass         | 256.90 emu yy | 7483   | 210    | 100.00 | 5.79   | 100.00 | 27.29  |
| 16.501   | Mass         | 256.90 emu UP | 11719  | 239    | 100.00 | 10.54  | 100.00 | 42.59  |
| 17.239   | Mass         | 256.90 emu yy | 5456   | 113    | 100.00 | 4.95   | 100.00 | 19.83  |
| 17.819   | Mass         | 321.90 amu UP | 4549   | 117    | 100.00 | 3.22   | 100.00 | 4.39   |
| 18.016   | Mass         | 256.90 emu PP | 6125   | 93     | 100.00 | 5.55   | 100.00 | 22.26  |
| 18.539   | Mass         | 321.90 emu BV | 15800  | 320    | 100.00 | 11.19  | 100.00 | 15.24  |
| + 18.368 | Mass         | 334.90 amu PP | 435914 | 7447   | 55.45  | 100.00 | 100.00 | 100.00 |
| + 18.967 | Mass         | 352.90 emu BP | 351201 | 5207   | 44.55  | 120.00 | 85.34  | 100.00 |
| 19.226   | Mass         | 321.90 amu UP | 103698 | 2163   | 100.00 | 73.46  | 100.00 | 100.00 |
| 20.520   | Mass         | 256.90 emu yy | 13359  | 263    | 100.00 | 12.43  | 100.00 | 18.55  |
| 20.549   | Mass         | 321.90 emu UP | 17120  | 258    | 45.34  | 12.15  | 55.35  | 16.51  |
| 20.552   | Mass         | 256.90 emu UP | 206537 | 273    | 54.55  | 15.74  | 100.00 | 75.00  |
| 21.175   | Mass         | 256.90 emu yy | 27614  | 305    | 100.00 | 24.98  | 100.00 | 100.00 |
| 21.429   | Mass         | 256.90 emu UP | 10924  | 154    | 100.00 | 9.32   | 100.00 | 32.70  |
| 21.542   | Mass         | 256.90 emu PY | 4374   | 53     | 100.00 | 5.87   | 100.00 | 15.39  |
| 21.812   | Mass         | 256.90 emu yy | 1195   | 24     | 100.00 | 1.08   | 100.00 | 4.35   |
| 21.946   | Mass         | 256.90 emu VE | 1339   | 34     | 100.00 | 1.22   | 100.00 | 4.37   |

85ENV620



## \*\*\* Area Percent \*\*\*

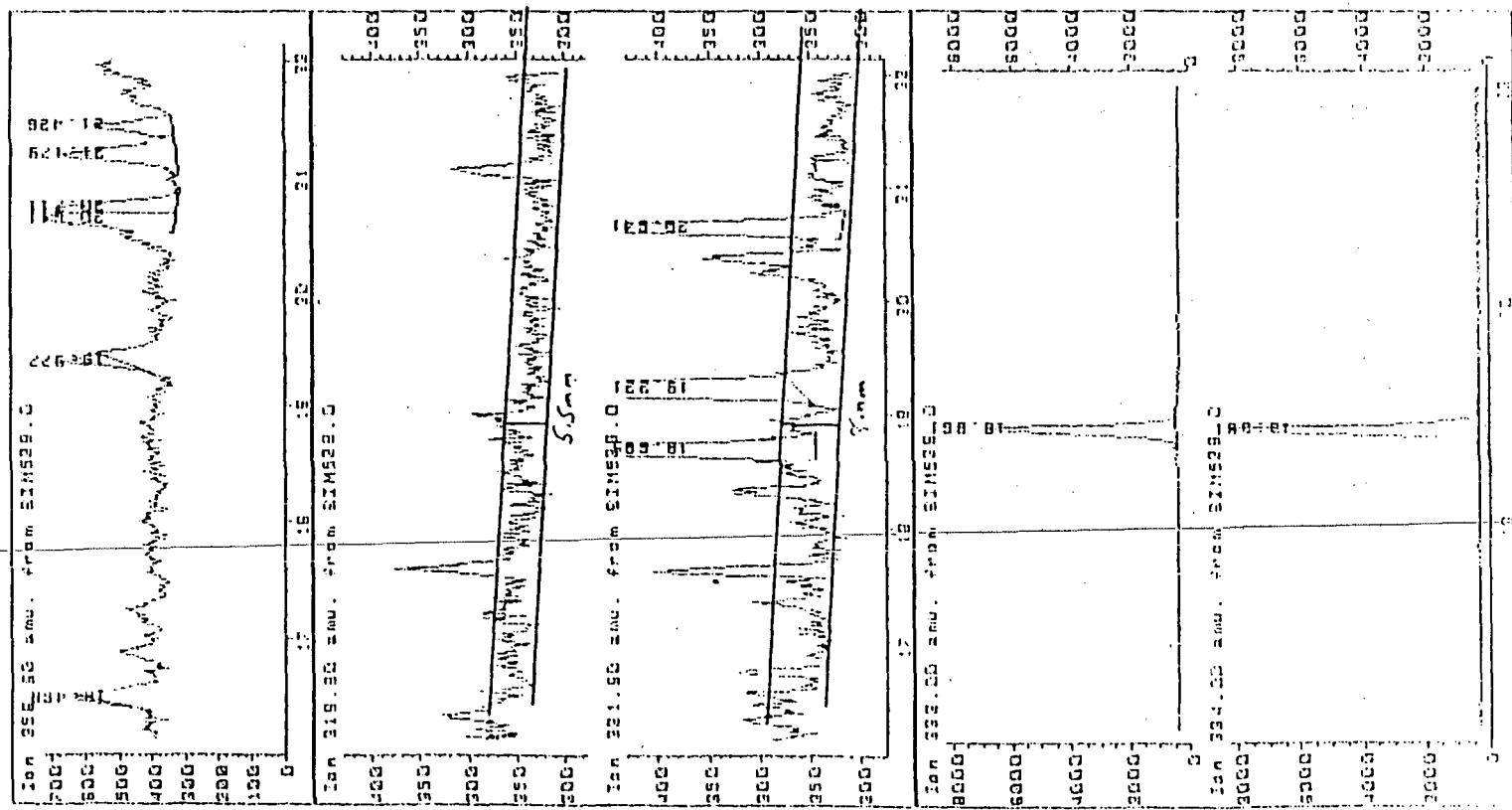
=====  
 ===== Report by Retention Time =====  
 =====

Operator: D. DEI ROSSI  
 Sample Info : 95ENUE22  
 Miss Info: SEDIMENT SAMPLE LOC. #5, 101.4g sample  
 Integration File Name : SIMS29.I

23 Sep 25 3:53 pm

| Ret Time | Signal Descr | Type          | Area   | Height | % Pk   | % SG   | % Lpk  | % Lsg  |
|----------|--------------|---------------|--------|--------|--------|--------|--------|--------|
| 16.463   | Mass         | 256.90 amu VU | 10141  | 240    | 100.00 | 5.17   | 100.00 | 20.50  |
| 15.502   | Mass         | 256.90 amu VP | 11586  | 254    | 100.00 | 7.26   | 100.00 | 38.79  |
| 16.394   | Mass         | 255.90 amu PP | 5753   | 117    | 100.00 | 3.21   | 100.00 | 18.27  |
| 17.517   | Mass         | 321.90 amu VP | 5556   | 173    | 100.00 | 3.22   | 100.00 | 4.45   |
| 17.536   | Mass         | 319.90 amu PP | 5535   | 121    | 100.00 | 5.52   | 100.00 | 100.00 |
| 16.572   | Mass         | 332.90 amu VU | 5743   | 136    | 100.00 | 1.81   | 100.00 | 1.84   |
| 16.525   | Mass         | 321.90 amu VP | 50172  | 605    | 100.00 | 11.51  | 100.00 | 15.03  |
| X 16.351 | Mass         | 334.90 amu PP | 45393  | 7953   | 35.55  | 100.00 | 100.00 | 100.00 |
| 16.961   | Mass         | 352.90 amu VP | 365759 | 5254   | 44.35  | 52.18  | 79.79  | 100.00 |
| 19.221   | Mass         | 321.90 amu PP | 187693 | 3829   | 100.00 | 72.24  | 100.00 | 100.00 |
| 19.377   | Mass         | 256.90 amu VU | 25205  | 298    | 100.00 | 15.25  | 100.00 | 31.93  |
| 20.364   | Mass         | 321.90 amu VP | 7240   | 132    | 100.00 | 2.78   | 100.00 | 3.83   |
| 20.444   | Mass         | 256.90 amu VU | 4541   | 140    | 100.00 | 2.76   | 100.00 | 13.19  |
| 20.475   | Mass         | 256.90 amu VU | 5356   | 153    | 100.00 | 2.34   | 100.00 | 12.84  |
| 20.511   | Mass         | 256.90 amu VP | 28899  | 430    | 100.00 | 13.19  | 100.00 | 165.63 |
| 20.541   | Mass         | 321.90 amu VP | 26315  | 123    | 100.00 | 15.14  | 100.00 | 14.20  |
| 20.711   | Mass         | 256.90 amu VP | 20004  | 356    | 100.00 | 12.17  | 100.00 | 82.91  |
| 21.154   | Mass         | 319.90 amu PP | 5459   | 99     | 100.00 | 45.18  | 100.00 | 57.13  |
| 21.179   | Mass         | 256.90 amu VU | 27477  | 340    | 100.00 | 15.72  | 100.00 | 21.13  |
| 21.125   | Mass         | 258.90 amu VP | 15055  | 174    | 100.00 | 7.94   | 100.00 | 47.33  |
| 21.342   | Mass         | 256.90 amu VU | 3216   | 97     | 100.00 | 2.19   | 100.00 | 17.31  |
| 21.378   | Mass         | 253.10 amu VP | 7511   | 131    | 100.00 | 1.13   | 100.00 | 1.13   |
| 21.430   | Mass         | 253.10 amu VP | 2102   | 15     | 100.00 | 0.03   | 100.00 | 0.03   |

SCENVERZ



## \*\*\* Area Percent \*\*\*

---

 ===== Report by Retention Time =====
 

---

Operator: D. DEI ROSSI

23 Sep 95 4:36 pm

Sample Info : 85ENV619 DUPLICATE

Misc Info: SEDIMENT SAMPLE LOC. #1, 33.0 g sample

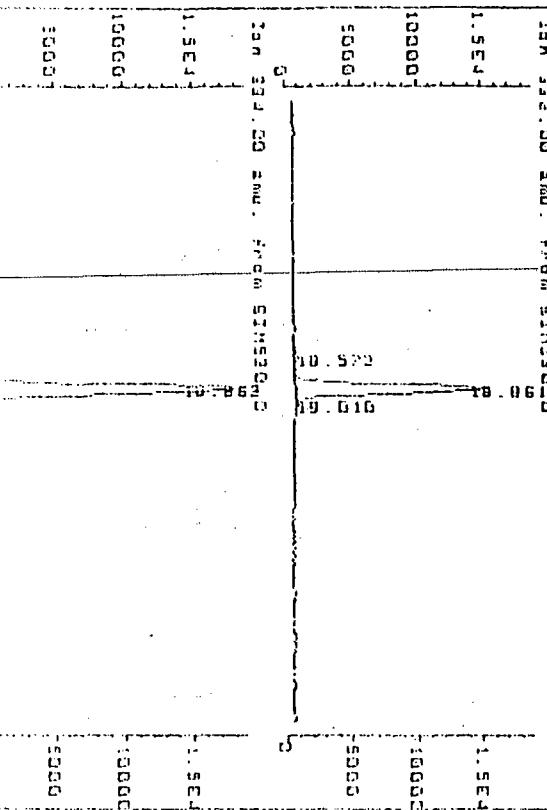
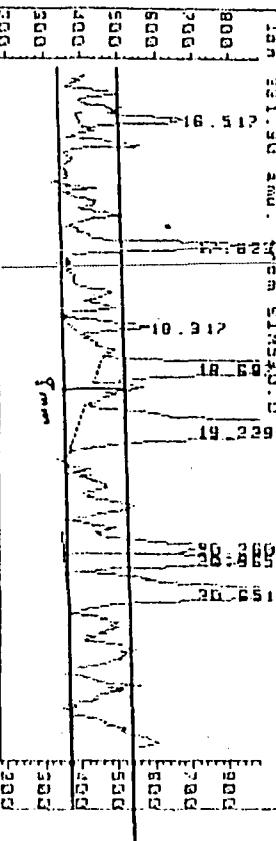
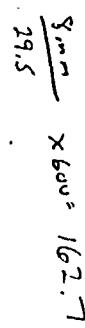
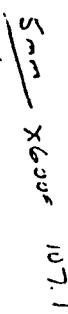
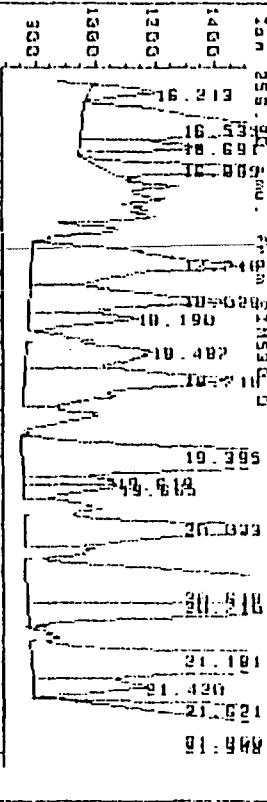
Integration File Name : SIME30.I

| Ret Time | Signal Descr | Type          | Area   | Height | % Pk   | % Sq  | % Lpk  | % Lsq |
|----------|--------------|---------------|--------|--------|--------|-------|--------|-------|
| 16.213   | Mass         | 256.90 amu BP | 10126  | 233    | 100.00 | 0.55  | 100.00 | 2.98  |
| 16.297   | Mass         | 321.90 amu PV | 5414   | 135    | 100.00 | 0.60  | 100.00 | 1.14  |
| 16.424   | Mass         | 321.90 amu UV | 3738   | 158    | 100.00 | 0.42  | 100.00 | 0.79  |
| 16.452   | Mass         | 332.00 amu VP | 1455   | 85     | 100.00 | 0.16  | 100.00 | 0.17  |
| 16.517   | Mass         | 321.90 amu UV | 16896  | 320    | 100.00 | 1.28  | 100.00 | 3.57  |
| 16.535   | Mass         | 256.90 amu PV | 102357 | 1043   | 100.00 | 5.53  | 100.00 | 30.16 |
| 16.631   | Mass         | 256.90 amu UV | 23126  | 451    | 100.00 | 1.26  | 100.00 | 6.91  |
| 16.747   | Mass         | 321.90 amu VP | 6193   | 189    | 100.00 | 0.91  | 100.00 | 1.73  |
| 16.885   | Mass         | 256.90 amu UV | 52999  | 1014   | 100.00 | 2.98  | 100.00 | 15.62 |
| 16.971   | Mass         | 256.90 amu UV | 5533   | 271    | 100.00 | 0.30  | 100.00 | 1.63  |
| 17.033   | Mass         | 256.90 amu UV | 16304  | 416    | 100.00 | 0.39  | 100.00 | 4.80  |
| 17.116   | Mass         | 321.90 amu UV | 1278   | 31     | 100.00 | 0.14  | 100.00 | 0.27  |
| 17.148   | Mass         | 256.90 amu UV | 22995  | 388    | 100.00 | 1.26  | 100.00 | 6.78  |
| 17.213   | Mass         | 256.90 amu UV | 5653   | 322    | 100.00 | 0.31  | 100.00 | 1.87  |
| 17.231   | Mass         | 321.90 amu UV | 4148   | 96     | 35.07  | 0.46  | 51.48  | 0.69  |
| 17.243   | Mass         | 256.90 amu UV | 5748   | 347    | 51.35  | 0.37  | 100.00 | 1.99  |
| 17.292   | Mass         | 256.90 amu UV | 16214  | 392    | 100.00 | 0.39  | 100.00 | 4.79  |
| 17.342   | Mass         | 321.90 amu UV | 3748   | 155    | 100.00 | 0.42  | 100.00 | 0.79  |
| 17.363   | Mass         | 321.90 amu UV | 4192   | 151    | 100.00 | 0.47  | 100.00 | 0.69  |
| 17.419   | Mass         | 256.90 amu VP | 13173  | 274    | 100.00 | 0.72  | 100.00 | 5.39  |
| 17.507   | Mass         | 321.90 amu VP | 27713  | 517    | 100.00 | 3.09  | 100.00 | 5.85  |
| 17.541   | Mass         | 256.90 amu P  | 17974  | 107    | 100.00 | 13.55 | 100.00 | 68.51 |
| 17.576   | Mass         | 256.90 amu P  | 30173  | 712    | 100.00 | 4.53  | 100.00 | 11.71 |

|         |      |        |     |    |         |       |        |       |        |        |
|---------|------|--------|-----|----|---------|-------|--------|-------|--------|--------|
| 16.150  | Mass | 255.30 | amu | VU | 22940   | 366   | 100.00 | 1.25  | 100.00 | 0.75   |
| 16.298  | Mass | 332.00 | amu | VU | 12778   | 158   | 100.00 | 1.41  | 100.00 | 0.50   |
| 18.317  | Mass | 321.90 | amu | VU | 12505   | 257   | 100.00 | 1.40  | 100.00 | 0.54   |
| 18.378  | Mass | 255.30 | amu | VU | 8782    | 221   | 100.00 | 0.48  | 100.00 | 0.59   |
| 18.487  | Mass | 255.30 | amu | VU | 42251   | 422   | 100.00 | 2.51  | 100.00 | 1.45   |
| 18.528  | Mass | 321.90 | amu | VU | 9330    | 140   | 100.00 | 0.77  | 100.00 | 1.46   |
| 18.572  | Mass | 332.00 | amu | VU | 20615   | 351   | 100.00 | 2.27  | 100.00 | 1.42   |
| 18.637  | Mass | 321.90 | amu | VU | 84103   | 1457  | 100.00 | 9.38  | 100.00 | 1.76   |
| 18.719  | Mass | 255.30 | amu | VU | 77154   | 577   | 100.00 | 4.21  | 100.00 | 2.72   |
| +18.861 | Mass | 332.00 | amu | VU | 350700  | 14768 | 97.22  | 93.76 | 100.00 | 0.00   |
| +18.961 | Mass | 321.90 | amu | VU | 15045   | 215   | 1.25   | 1.78  | 1.00   | 2.58   |
| 18.947  | Mass | 319.90 | amu | VU | 8245    | 122   | 0.94   | 15.26 | 0.95   | 42.17  |
| +18.862 | Mass | 334.00 | amu | VU | 1061144 | 15316 | 100.00 | 98.25 | 100.00 | 100.00 |
| 19.010  | Mass | 332.00 | amu | VU | 22358   | 232   | 54.33  | 2.46  | 100.00 | 2.83   |
| 19.024  | Mass | 255.30 | amu | VU | 18345   | 248   | 45.07  | 1.06  | 93.02  | 5.41   |
| 19.086  | Mass | 334.00 | amu | VU | 4057    | 104   | 100.00 | 0.38  | 100.00 | 0.39   |
| 19.229  | Mass | 321.90 | amu | VU | 473483  | 6633  | 100.00 | 52.82 | 100.00 | 0.00   |
| 19.395  | Mass | 255.30 | amu | VU | 216293  | 2206  | 100.00 | 11.61 | 100.00 | 63.72  |
| 19.510  | Mass | 321.90 | amu | VU | 11504   | 515   | 100.00 | 0.35  | 100.00 | 2.59   |
| 19.665  | Mass | 256.30 | amu | VU | 15839   | 329   | 100.00 | 0.87  | 100.00 | 4.87   |
| 19.729  | Mass | 321.90 | amu | VU | 7235    | 120   | 100.00 | 0.31  | 100.00 | 1.55   |
| 19.857  | Mass | 255.30 | amu | VU | 16608   | 256   | 100.00 | 0.31  | 100.00 | 4.89   |
| 20.023  | Mass | 256.30 | amu | VU | 91515   | 1262  | 100.00 | 4.66  | 100.00 | 2.23   |
| 20.087  | Mass | 321.90 | amu | VU | 8488    | 146   | 152.56 | 9.33  | 100.00 | 0.05   |
| 20.132  | Mass | 319.90 | amu | VU | 1507    | 161   | 17.53  | 1.16  | 90.47  | 1.15   |
| 20.201  | Mass | 255.30 | amu | VU | 1101    | 231   | 100.00 | 0.32  | 100.00 | 0.36   |
| 20.250  | Mass | 321.90 | amu | VU | 7249    | 276   | 100.00 | 7.03  | 100.00 | 0.75   |
| 20.383  | Mass | 321.90 | amu | VU | 31569   | 152   | 100.00 | 5.16  | 100.00 | 0.33   |
| 20.531  | Mass | 321.90 | amu | VU | 31561   | 213   | 100.00 | 1.56  | 100.00 | 0.30   |

|         |      |        |        |        |      |        |       |        |        |
|---------|------|--------|--------|--------|------|--------|-------|--------|--------|
| 21. 822 | Mass | 334.00 | vp     | 6529   | 126  | 100.00 | 0.50  | 100.00 | 0.30   |
| 21. 151 | Mass | 321.90 | smu vu | 26539  | 169  | 52.38  | 2.28  | 100.00 | 4.34   |
| 21. 159 | Mass | 319.90 | smu vp | 19582  | 301  | 47.51  | 29.55 | 90.37  | 100.00 |
| 21. 181 | Mass | 256.90 | smu vu | 276340 | 3475 | 100.00 | 15.38 | 100.00 | 31.12  |
| 21. 420 | Mass | 256.90 | smu vp | 25237  | 384  | 100.00 | 1.27  | 100.00 | 5.65   |
| 21. 474 | Mass | 321.90 | smu vp | 17485  | 204  | 100.00 | 1.95  | 100.00 | 3.69   |
| 21. 821 | Mass | 255.90 | smu pp | 61035  | 1010 | 100.00 | 3.33  | 100.00 | 17.38  |
| 21. 788 | Mass | 321.90 | smu py | 1241   | 46   | 100.00 | 0.15  | 100.00 | 0.50   |
| 21. 835 | Mass | 321.90 | smu vp | 1501   | 54   | 100.00 | 0.17  | 100.00 | 0.32   |
| 21. 864 | Mass | 319.90 | smu pp | 2029   | 57   | 100.00 | 3.22  | 100.00 | 10.37  |
| 21. 900 | Mass | 256.90 | smu py | 21572  | 557  | 100.00 | 1.18  | 100.00 | 8.41   |
| 21. 948 | Mass | 256.90 | smu vb | 2873   | 153  | 100.00 | 2.16  | 100.00 | 0.85   |
| 21. 953 | Mass | 319.90 | smu ps | 1382   | 49   | 100.00 | 2.20  | 100.00 | 7.41   |

85ENV619 DUPLICATE



۱۷۰

۱۷۵

## \*\*\* Area Percent \*\*\*

===== Report by Retention Time =====

Operator: D. DEI ROSSI

23 Sep 85 5:22 pm

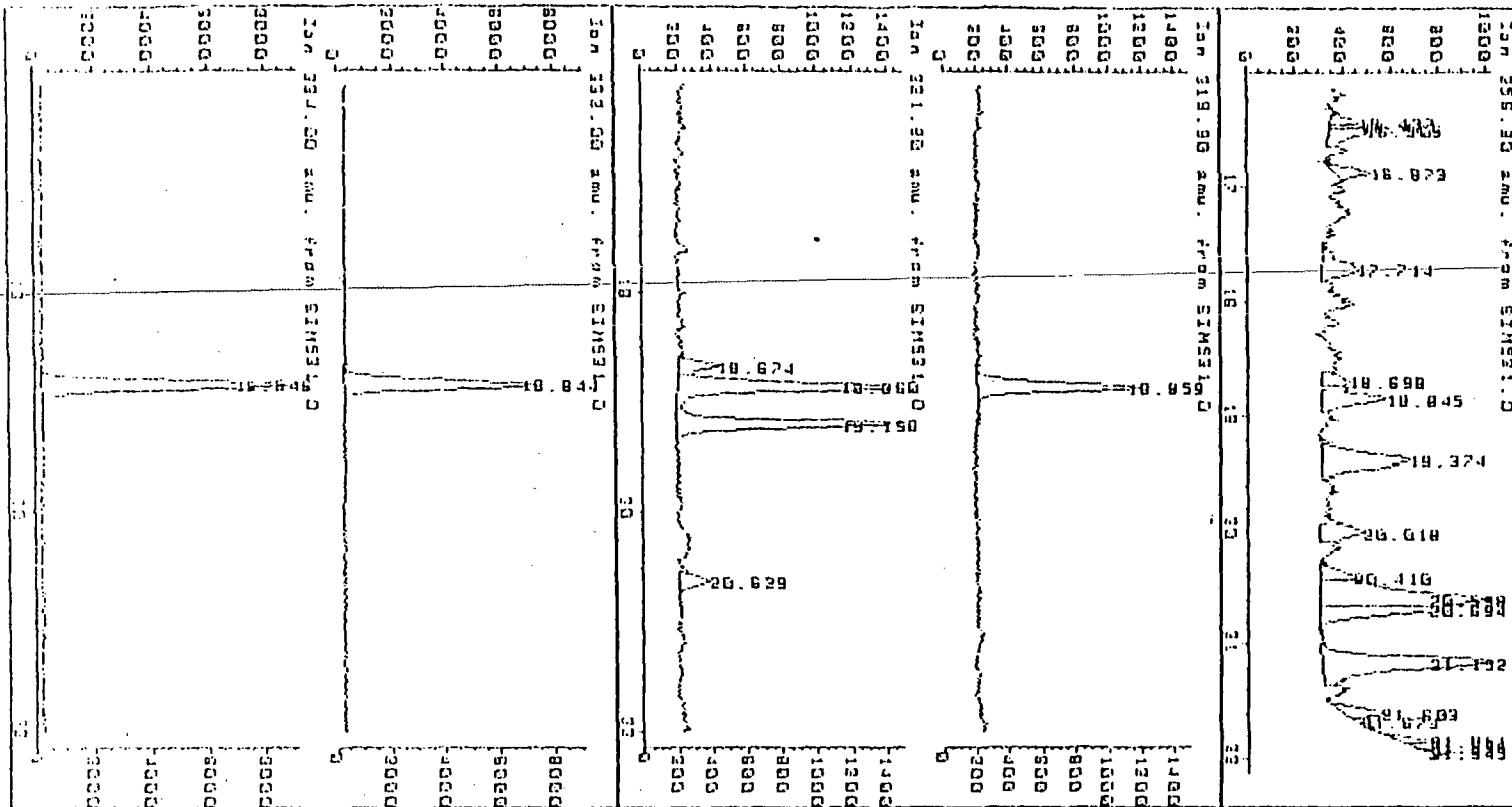
Sample Info : STANDARD ADDITION; 1.5795 mg native TCDD

Misc Info: SEDIMENT SAMPLE LOC. #5, 104.9g sample

Integration File Name : SIMSET1.I

| Ret Time | Signal Descr | Type          | Area   | Height | % Pk   | % Sq   | % Lpk  | % Lsg  |
|----------|--------------|---------------|--------|--------|--------|--------|--------|--------|
| 16.437   | Mass         | 256.90 amu UV | 3980   | 119    | 100.00 | 1.44   | 100.00 | 6.36   |
| 16.471   | Mass         | 256.90 amu UV | 2370   | 136    | 100.00 | 0.86   | 100.00 | 3.92   |
| 16.505   | Mass         | 256.90 amu UV | 10050  | 163    | 100.00 | 3.65   | 100.00 | 16.83  |
| 16.573   | Mass         | 256.90 amu PP | 8310   | 155    | 100.00 | 2.29   | 100.00 | 10.43  |
| 17.714   | Mass         | 256.90 amu BV | 13430  | 149    | 100.00 | 4.87   | 100.00 | 22.20  |
| 18.674   | Mass         | 321.90 amu UV | 12640  | 239    | 100.00 | 7.52   | 100.00 | 16.62  |
| 18.698   | Mass         | 256.90 amu UV | 5400   | 117    | 100.00 | 1.95   | 100.00 | 9.94   |
| → 18.846 | Mass         | 334.00 amu PP | 504310 | 6817   | 51.77  | 100.00 | 100.00 | 100.00 |
| → 18.844 | Mass         | 332.00 amu PP | 397209 | 6266   | 40.78  | 100.00 | 79.72  | 100.00 |
| → 18.859 | Mass         | 319.90 amu PP | 55531  | 980    | 5.71   | 100.00 | 11.02  | 100.00 |
| → 18.845 | Mass         | 256.90 amu UV | 16964  | 292    | 1.74   | 6.15   | 3.36   | 28.04  |
| → 18.860 | Mass         | 321.90 amu UV | 76050  | 1240   | 100.00 | 45.84  | 100.00 | 100.00 |
| 19.198   | Mass         | 321.90 amu VP | 66674  | 1285   | 100.00 | 40.18  | 100.00 | 87.37  |
| 19.374   | Mass         | 256.90 amu UV | 35410  | 375    | 100.00 | 12.35  | 100.00 | 56.53  |
| 20.019   | Mass         | 256.90 amu UV | 12080  | 181    | 100.00 | 4.39   | 100.00 | 19.89  |
| 20.410   | Mass         | 256.90 amu PV | 6484   | 141    | 100.00 | 2.35   | 100.00 | 10.73  |
| 20.589   | Mass         | 256.90 amu UV | 80464  | 711    | 100.00 | 21.35  | 100.00 | 100.00 |
| 20.629   | Mass         | 321.90 amu VP | 10555  | 182    | 100.00 | 8.38   | 100.00 | 13.86  |
| 20.684   | Mass         | 256.90 amu VP | 27763  | 511    | 100.00 | 10.07  | 100.00 | 45.88  |
| 21.152   | Mass         | 256.90 amu UV | 58537  | 707    | 100.00 | 29.51  | 100.00 | 83.48  |
| 21.603   | Mass         | 256.90 amu PV | 7357   | 180    | 100.00 | 2.67   | 100.00 | 12.16  |
| 21.673   | Mass         | 256.90 amu VP | 1415   | 11     | 100.00 | 0.51   | 100.00 | 0.74   |
| 21.751   | Mass         | 256.90 amu PP | 4894   | 80     | 100.00 | 1.37   | 100.00 | 7.30   |
| 21.765   | Mass         | 256.90 amu V  | 101    | 11     | 100.00 | 1.02   | 100.00 | 4.54   |

STD ADDITION





File Spring River Fish CC: Kurt Stormer  
& Sediment plan David Fussman

## MISSOURI DEPARTMENT OF CONSERVATION

Fish and Wildlife Research Center • 1110 College Avenue  
Columbia, Missouri 65201 • Phone 314/449-3761  
LARRY R. GALE, Director

October 9, 1985

Syntex Research  
3401 Hillview Ave.  
Palo, California 94303

Attention Dr. Throop

Dear Dr. Throop:

I recently received the revised data tables from the 1984 Spring River fish samples. I suggest additional calculations be made to determine whole body concentrations of TCDD for sites one through five. The fillet yield from this year's samples could be substituted for the absent values.

The percent of fillet tissue removed from whole fish this year was 0.15, 0.18, 0.15, 0.12, and 0.15 from sites one through five, respectively. The average of these numbers (0.15) would be satisfactory for calculation purposes. I feel confident these values will be repeatable from year-to-year. There is little variation within a species in this characteristic, especially from fish taken from the same location during the same month of the year. We routinely predict similar characteristics of fish from length measurements with narrow statistical confidence limits.

Although these additional data points are not required for the statistical analysis they will be useful for year-to-year comparisons. By incorporating these new figures, the whole body values originally reported will be revised downward, but not to a great degree. The maximum reduction would be less than 20 percent.

This year's samples were received in good order by the University of Nebraska laboratory. However, there was some concern about tissue preparation. I was somewhat surprised that this problem was not resolved since a similar concern was expressed last year. We do not have the equipment to grind fish at our lab. We could have them prepared at our contract laboratory at the University of Missouri, but would request the cost be picked up by Syntex. I would still prefer the tissue grinding be done at the analytical laboratory to reduce handling of the samples.

### COMMISSION

JEFF CHURAN  
Chillicothe

JOHN POWELL  
Rolla

JOHN B. MAHAFFEY  
Springfield

RICHARD T. REED  
East Prairie

Dr. Throop

-2-

October 9, 1985

I hope there is no further confusion over labeling of the samples. The labels are simply identification tags that refer to the attached data sheets which describe the manner in which the samples are to be analyzed according to the agreed upon protocol. The "whole body" designation on B sample identification tags last year referred to the calculated whole body values which were to be derived from analysis of this portion of fish as indicated in the memo which accompanied the fish and as specified in the Verona fish and sediment sampling plan. There was only one sample of fish (site 1) that was to be left whole and analyzed.

Please let me know if I can be of any further assistance.

Sincerely,



Ronald L. Crunkilton  
Water Quality Research Biologist

RLC:tja

cc: Mr. K. S. Ritchey  
Mr. D. Robertson

RECEIVED  
OCT 18 1985  
DR. L. THROOP